

2007

COMMUNITY REFERENCE LABORATORY (CRL)
WORKSHOP AND TRAINING COURSE FOR
PESTICIDE RESIDUES IN FOOD

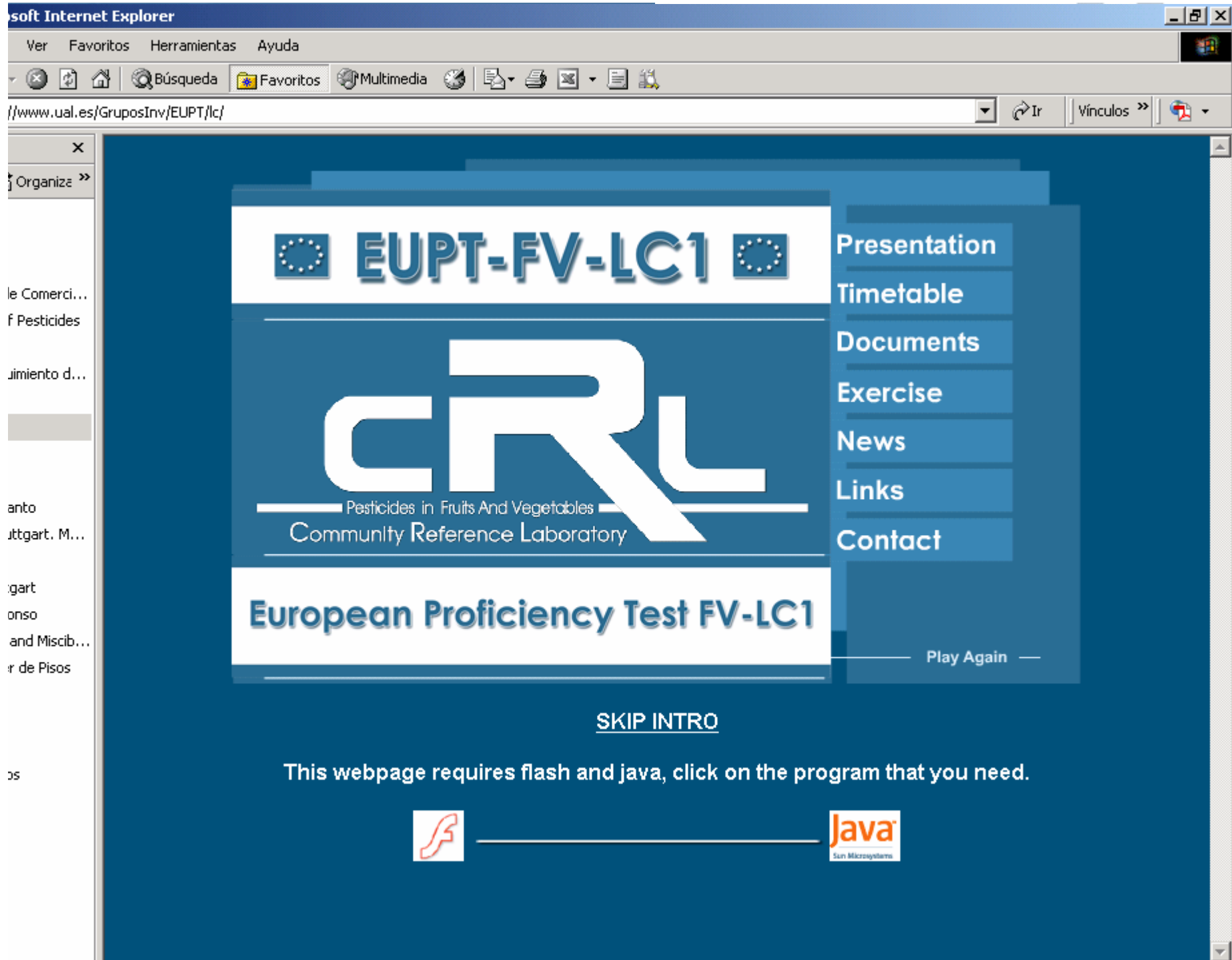
26-29 September 2007
Valencia – SPAIN



European Proficiency Test Low Concentration EUPT-LC1

Amadeo R. Fernández-Alba





2007

COMMUNITY REFERENCE LABORATORY (CRL)
WORKSHOP AND TRAINING COURSE FOR
PESTICIDE RESIDUES IN FOOD

26-29 September 2007
Valencia – SPAIN

ORGANISING TEAM at CRL-University of Almeria:

Dr. Amadeo R. Fernández-Alba. senior Chemist.
Ms. Paula Medina. Chemist.
Mr. Octavio Malato. Chemist.
Mr. Antonio Valverde Monterreal (Jr.). Chemist.

University of Almeria
University of Almeria
University of Almeria
University of Almeria

ADVISORY GROUP:

Mr. Arne Andersson. Head of Division.
Dr. Antonio Valverde García. Senior Chemist.
Dr. Carmelo Rodríguez. Senior Chemist.
Dr. Miguel Gamón. Senior Chemist.
Dr. Michlangelo Anastassiades. Senior Chemist.
Dr. Mette Erecius Poulsen. Senior Chemist
Dr. André de Kok. Senior Chemist.
Dr. Tuija Pihlström. Senior Chemist.
Mr. Stewart Reynolds. senior Chemist.
Dr. Ralf Lippold. Senior Chemist.

NFA. Uppsala. Sweden
University of Almeria. Spain
University of Almeria. Spain
Generalitat Valenciana. Spain
CVUA Stuttgart. Germany
Danish Institute for Food and Veterinary Research
VWA. Amsterdam. The Netherlands
NFA. Uppsala. Sweden
CSL. York. United Kingdom
CVUA Freiburg. Germany



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Co-Head of CRL-FV

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COMMUNITY REFERENCE LABORATORY (CRL)
WORKSHOP AND TRAINING COURSE FOR
PESTICIDE ANALYSIS

26-29 September
Valencia



European Proficiency Test FV-LC1.



- Presentation

- Timetable

- Documents

- Exercise

- News

- Links

- Contact

... Play Again ...

TIMETABLE

Activity

Date

Deadline for receiving Application Form from invited laboratories

16th of March 2007 ✓

Sample Treatment, Homogenisation
Storage/Stability Test

March 2007 ✓

Deadline for the Payment of Shipping Costs
and/or demonstration that payment
procedure has been initiated

30th of March 2007 ✓

Sample Distribution.

9th-12th of April 2007 ✓

Deadline for receiving Form 1

13th of April 2007 ✓

Deadline for receiving results Forms 2 and 3

7th of May 2007 ✓

Preliminary Report: **only results, no statistical
treatment.**

July 2007 ✓

Final Report

December 2007



Z-ALBA
Co-ordinator of CRL-FV

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26-29 Sep
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European Proficiency Test FV-LC1



- Presentation

- Timetable

- Documents included here.

- Exercise

- News

- Links

- Contact

... Play Again ...

DOCUMENTS

Application Form



Word
Version

Pesticide List



Word
Version

e to obtain the FORMS

[Click Here](#)

You need your password to e



Z-ALBA
EU Field of CRL-FV

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PESTICIDE RESIDUES IN FOOD

26-29 September
Valencia – Spain



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The Pesticide List had 46 possible pesticides that could be present in the sample. Pesticides were to be reported with no parent compounds and/or no transformation products

12 pesticides were used to treat the sample

A MPRL of 0.005 mg/Kg was fixed to all the pesticides



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PESTICIDE R26-29 Septe
Valencia -

Pesticides	MPRL	Pesticides	MPRL
Acephate	0.005	Imidacloprid (only parent compound)	0.005
Acetamiprid	0.005	Iprodione	0.005
Acrinathrin	0.005	Kresoxim-methyl	0.005
Azoxystrobin	0.005	Lambda-cyhalothrin	0.005
Bifenthrin	0.005	Methamidophos	0.005
Bromopropylate	0.005	Monocrotophos	0.005
Carbaryl	0.005	Myclobutanil	0.005
Chlorothalonil	0.005	Omethoate (expressed as Omethoate)	0.005
Chlorpyrifos	0.005	Oxamyl (expressed as Oxamyl)	0.005
Chlorpyrifos-methyl	0.005	Oxydemeton-methyl (expressed as Oxydemeton-methyl)	0.005
Cypermethrin	0.005	Parathion	0.005
Cyprodinil	0.005	Pirimicarb	0.005
Deltamethrin	0.005	Pirimiphos-methyl	0.005
Diazinon	0.005	Prochloraz	0.005
Demeton-S-Methyl (expressed as Demeton-S-Methyl)	0.005	Procymidone	0.005
Demeton-S-methyl sulfone (expressed as Demeton-S-Methyl sulfone)	0.005	Propiconazole	0.005
Dichlofluanid	0.005	Pyrimethanil	0.005
Dichlorvos	0.005	Tebuconazole	0.005
Dicofol	0.005	Tetraconazole	0.005
Dimethoate (expressed as Dimethoate)	0.005	Thiophanate-methyl	0.005
Fenhexamid	0.005	Tolyfluanid	0.005
Fenitrothion	0.005	Vinclozolin (only parent compound)	0.005
Fludioxonil	0.005		0.005
Imazalil	0.005		

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Sample treatment



The pesticides were spiked as a standard solution to 10 kg of pears



To have low levels, the 10 kg are
diluted with no treated 60kg



EUROPEAN UNION
EUROPEAN COMMISSION
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**Mixed and
Chopped**



Minced



Frozen



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Homogeneise



Packed



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Participants



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Countries	No of Labs	NRLs	In EUPT-FV9 in Categ A
AUSTRIA	1	Yes	TOTAL 23 (76%)
BELGIUM	1	Yes	
BULGARY	1	Yes	
CYPRUS	1	Yes	
CZECH REPUBLIC	1	Yes	
FINLAND	1	Yes	
FRANCE	1	Yes	
GERMANY	9	1 Yes 8 No	
GREECE	1	Yes	
HUNGARY	1	Yes	
IRELAND	1	Yes	
ITALY	1	Yes	
LATVIA	1	Yes	
POLAND	1	Yes	
PORTUGAL	1	Yes	
SLOVAKIA	1	Yes	
SPAIN	2	1 Yes 1 No	
SWEDEN	2	1 Yes 1 No	
THE NETHERLANDS	1	Yes	
UNITED KINGDOM	1	Yes	
TOTAL	30	20	

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RESULTS



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PESTICIDES	Median (mg/Kg)	Nº of reported results	Nº of NA (not sought)	False negatives	% of results from the total 30
Acetamiprid	0.033	26	1	1	87
Carbaryl	0.026	26	2	0	87
Diazinon	0.024	28	0	0	93
Dimethoate	0.024	27	0	1	90
Imazalil	0.026	22	3	3	73
Imidacloprid	0.029	24	2	2	80
Iprodione	0.025	22	1	6	73
Omethoate	0.021	26	1	1	87
Oxydemeton-methyl	0.024	18	7	3	60
Pyrimethanil	0.023	25	2	1	83
Tetraconazole	0.029	25	2	1	83
Total		269	21 (8%)	19 (6,7%)	

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WORKSHOP AND TRAINING COURSE FOR
PESTICIDE RESIDUES IN FOOD**EUPT-FV9**

PESTICIDES	Median (mg/Kg)	N° of reported results	N° of NA (not sought)	False negatives	% of results from the total 132
Bupirimate	0.790	110	20	2	83
Cyprodinil	0.954	115	15	2	87
Diazinon	0.272	130	2	0	98
Endosulfan α	0.411	124	4	0	95
Endosulfan β	0.326	124	4	0	95
Fenhexamid	0.338	96	29	7	73
Fenitrothion	0.791	123	7	2	93
Fludioxonil	0.516	103	28	1	78
Hexythiazox	1.05	71	60	1	54
Iprodione	0.725	117	8	7	89
Myclobutanil	0.759	118	13	1	89
Oxamyl	0.756	111	15	6	84
Penconazole	0.486	116	14	2	88
Procymidone	0.606	129	3	0	98
Pyrimethanil	0.880	116	15	1	88
Quinoxifen	1.03	90	41	1	68
Tebuconazole	0.800	107	22	3	81
Tolyfluanid	1.60	118	14	0	89
Triadimenol	1.32	100	27	5	76
TOTAL		81%	14%	2%	

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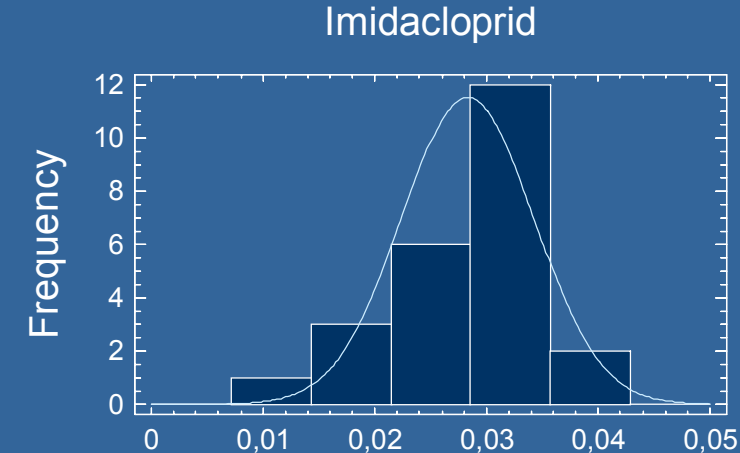
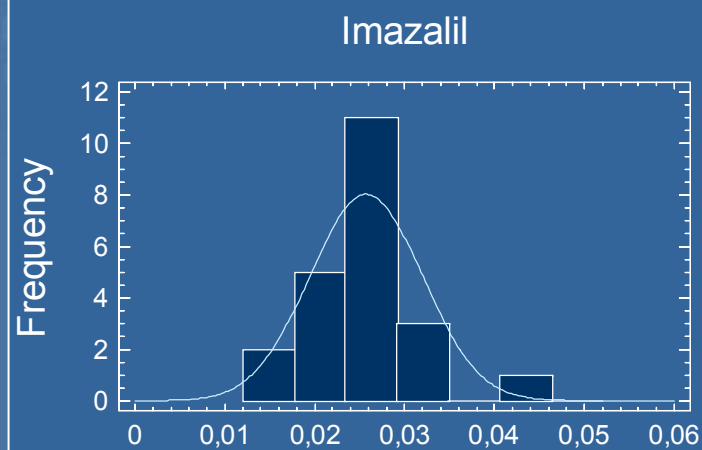
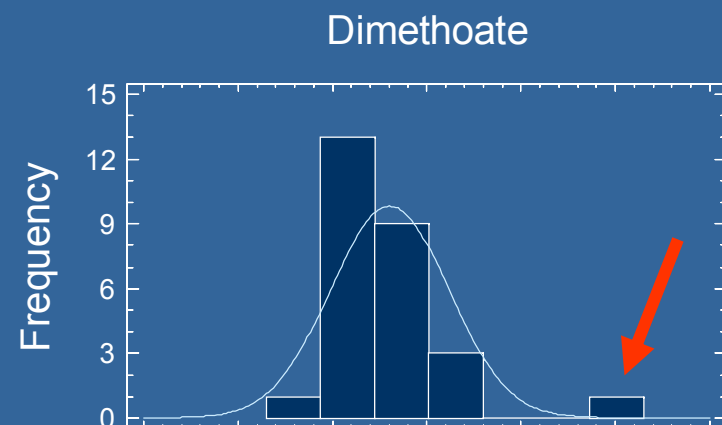
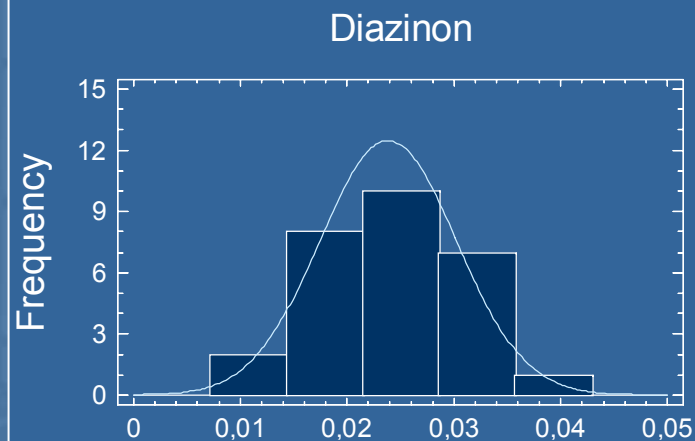
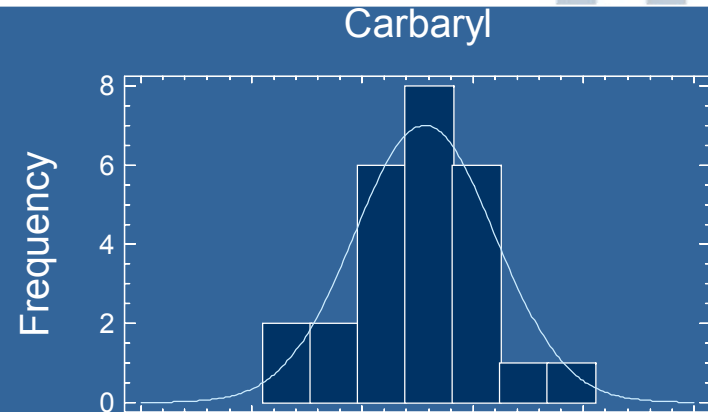
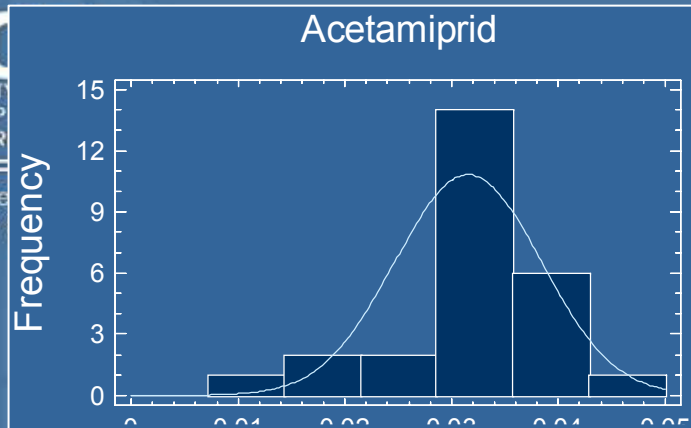
EU PT08



PESTICIDES	Nº of reported results	Nº of NA (not sought)	False negatives	% of results from the total 128
Acetamiprid	78	47	3	61
Azoxystrobin	113	15	0	88
Bifenthrin	119	9	0	93
Bromopropylate	125	3	0	98
Carbaryl	108	17	3	84
Carbendazim	94	34	0	74
Chlorpyrifos	127	1	0	99
Cyprodinil	114	13	1	89
Diazinon	127	1	0	99
Dichlofluanid	113	5	10	88
Fludioxonil	92	33	3	72
Imazalil	107	14	7	84
Lambda-cyhalothrin	121	5	2	95
Myclobutanil	114	14	0	89
Parathion	114	10	4	89
Pirimicarb	114	14	0	89
Total	1824	235 (12%)	31(1,7%)	



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Co-Head of CRL-FV

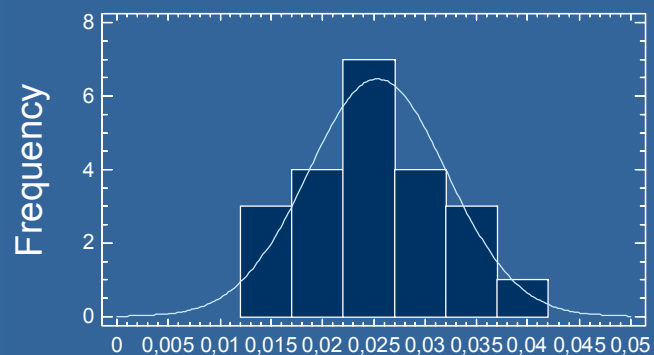


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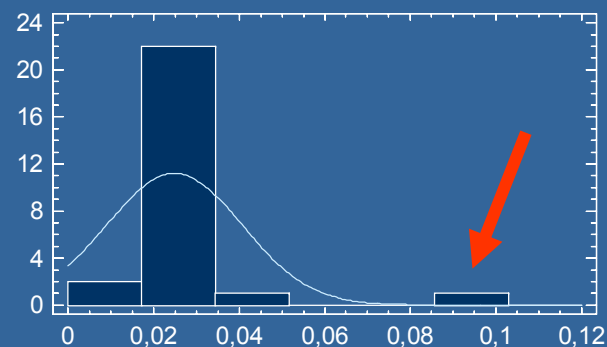
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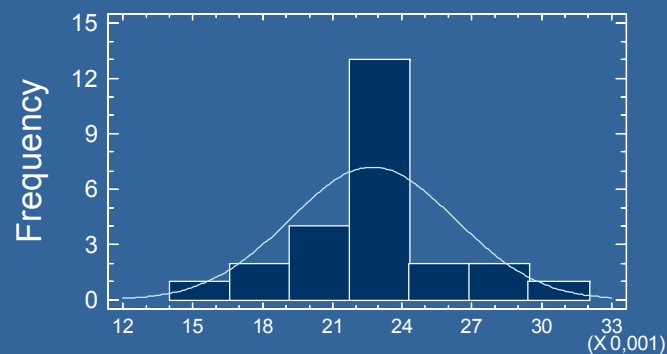
Iprodione



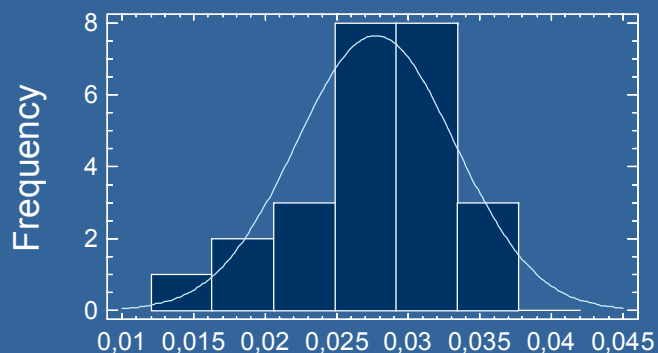
Omethoate



Pyrimethanil



Tetraconazole



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26-29 September 2007

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Iprodione False Negatives



Lab Code	Scope	Solvent or Matrix	Extraction Solvent	Clean up Step	Determination
EUPT-FV-LC1-005	ND	M	Acetone/ Ethyl Acetate/Hexane		GC-MS
EUPT-FV-LC1-009	0.024	M	Acetone/ Dichloromethane	GPC	GC-MS
EUPT-FV-LC1-016	ND	M	Acetonitrile		GC-ECD
EUPT-FV-LC1-018	ND	M	Acetonitrile		GC-MS
EUPT-FV-LC1-020	ND	M	Ethyl Acetate	GPC	GC-ECD
EUPT-FV-LC1-025	0.024	M	Acetone/Dichloromethane/ Petroleum Ether		GC-MS
EUPT-FV-LC1-026	ND	S	Ethyl Acetate		GC-FPD

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False Positives



	Chlorothalonil	Dichlofluanid	Dicofol	Procyimdone	Chlorpyrifos- methyl
EUPT-FV-LC1-018		0.042	0.039	0.045	
EUPT-FV-LC1-026					0.036



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EUPT-FV-LC1 z-scores



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z-Score

$$Z = x - \mu / \sigma$$

μ : Assigned value (median*)

σ : Standard deviation (25%)

$$|z| \leq 2$$

Acceptable

$$2 \leq |z| \leq 3$$

Questionable

$$|z| > 3$$

Unacceptable

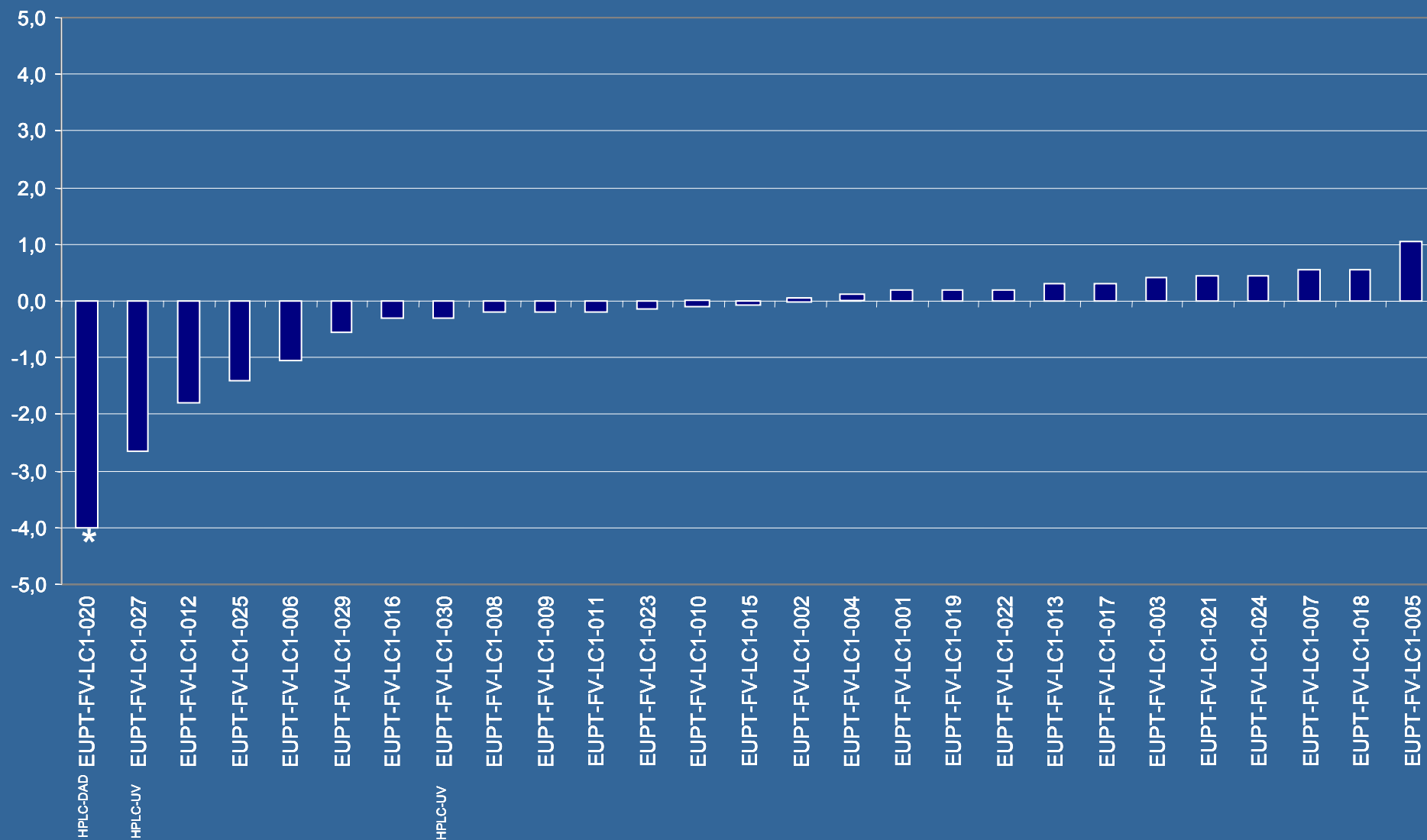


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GC
LC
GC and LC

* False Negatives

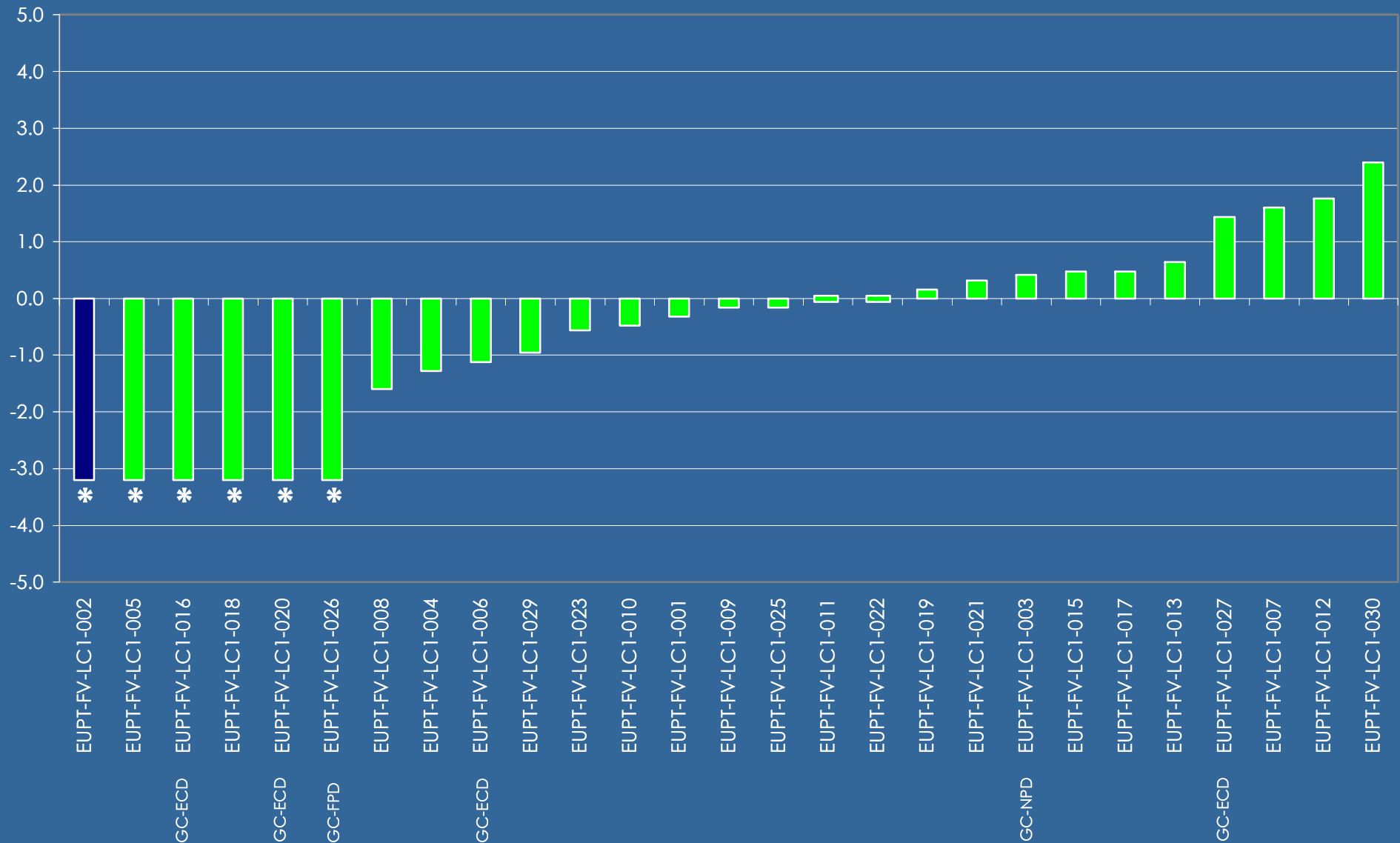
Acetamiprid



GC
LC
GC and LC

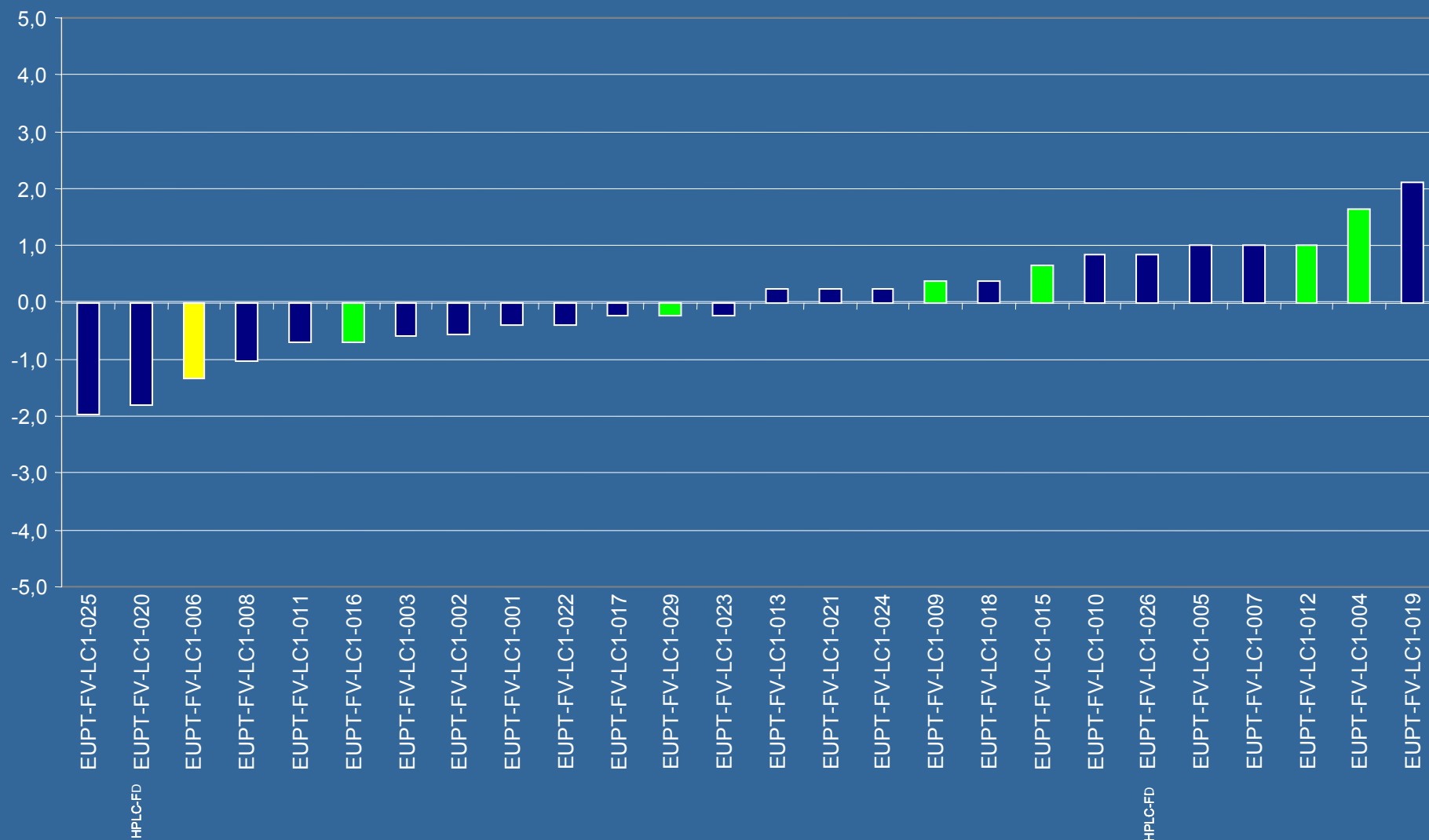
* False Negatives

Iprodione



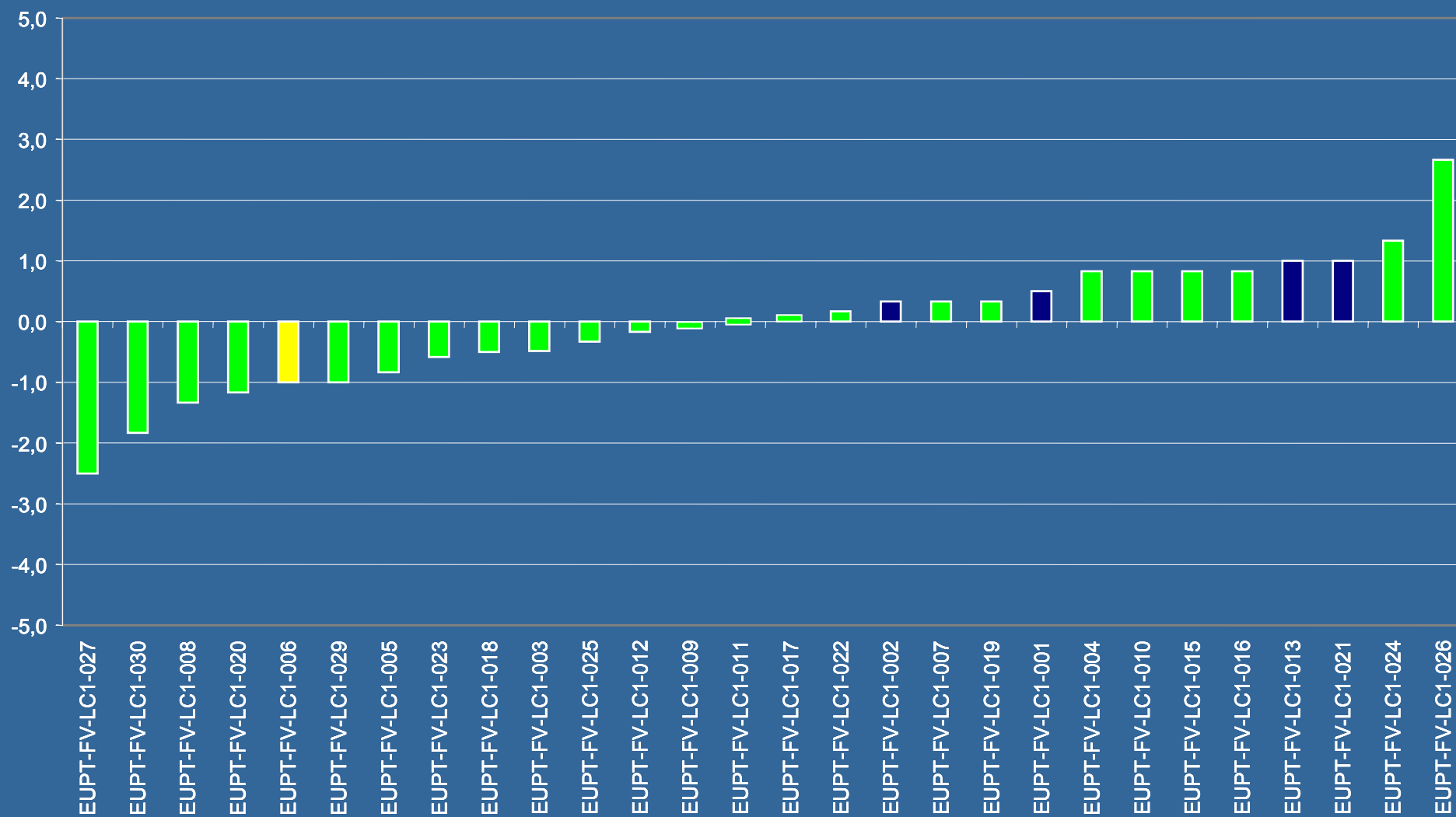
- GC
 - LC
 - GC and LC
- * False Negatives

Carbaryl



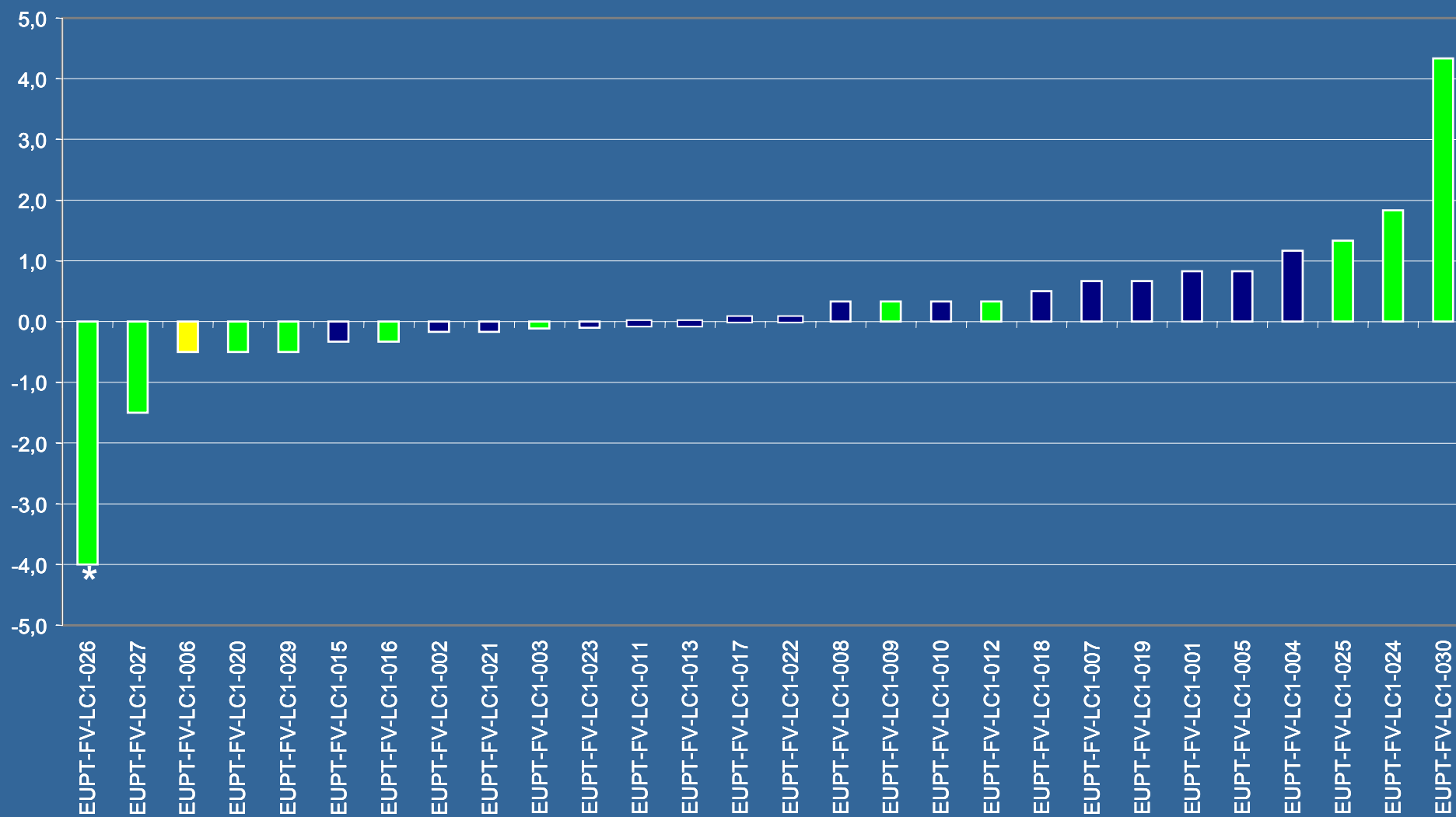
GC * False Negatives
LC
GC and LC

Diazinon



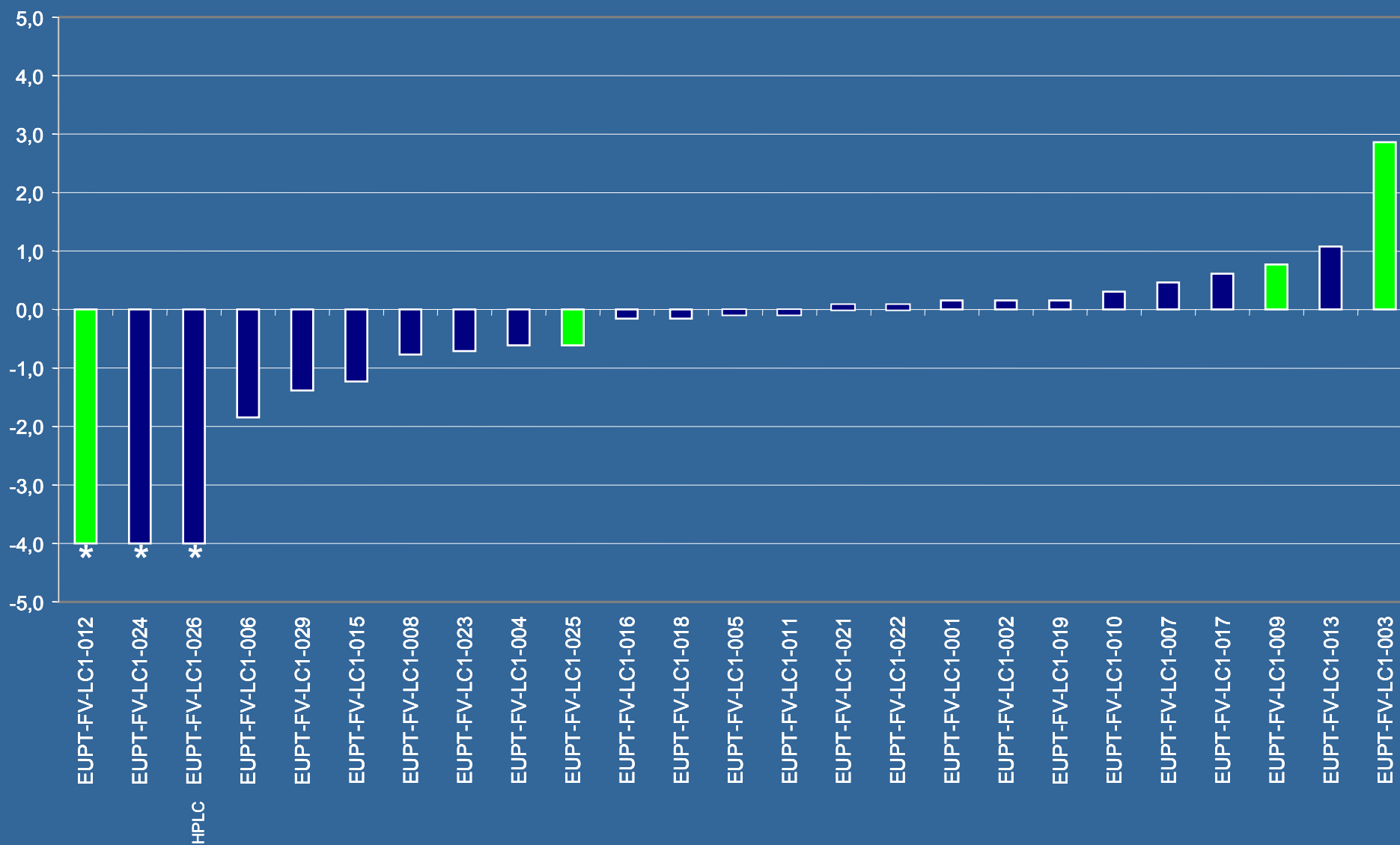
GC * False Negatives
LC
GC and LC

Dimethoate



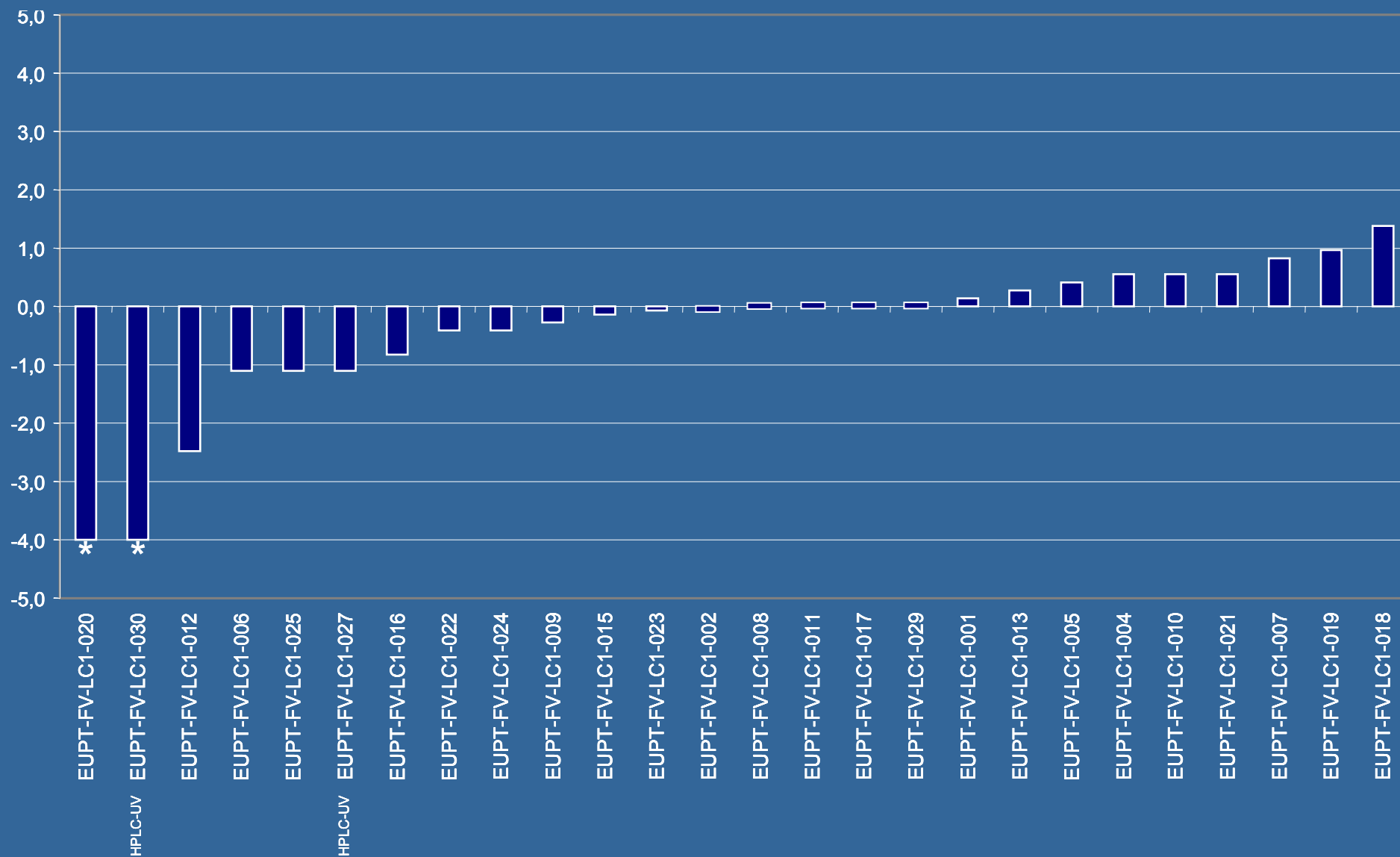
GC * False Negatives
LC
GC and LC

Imazalil



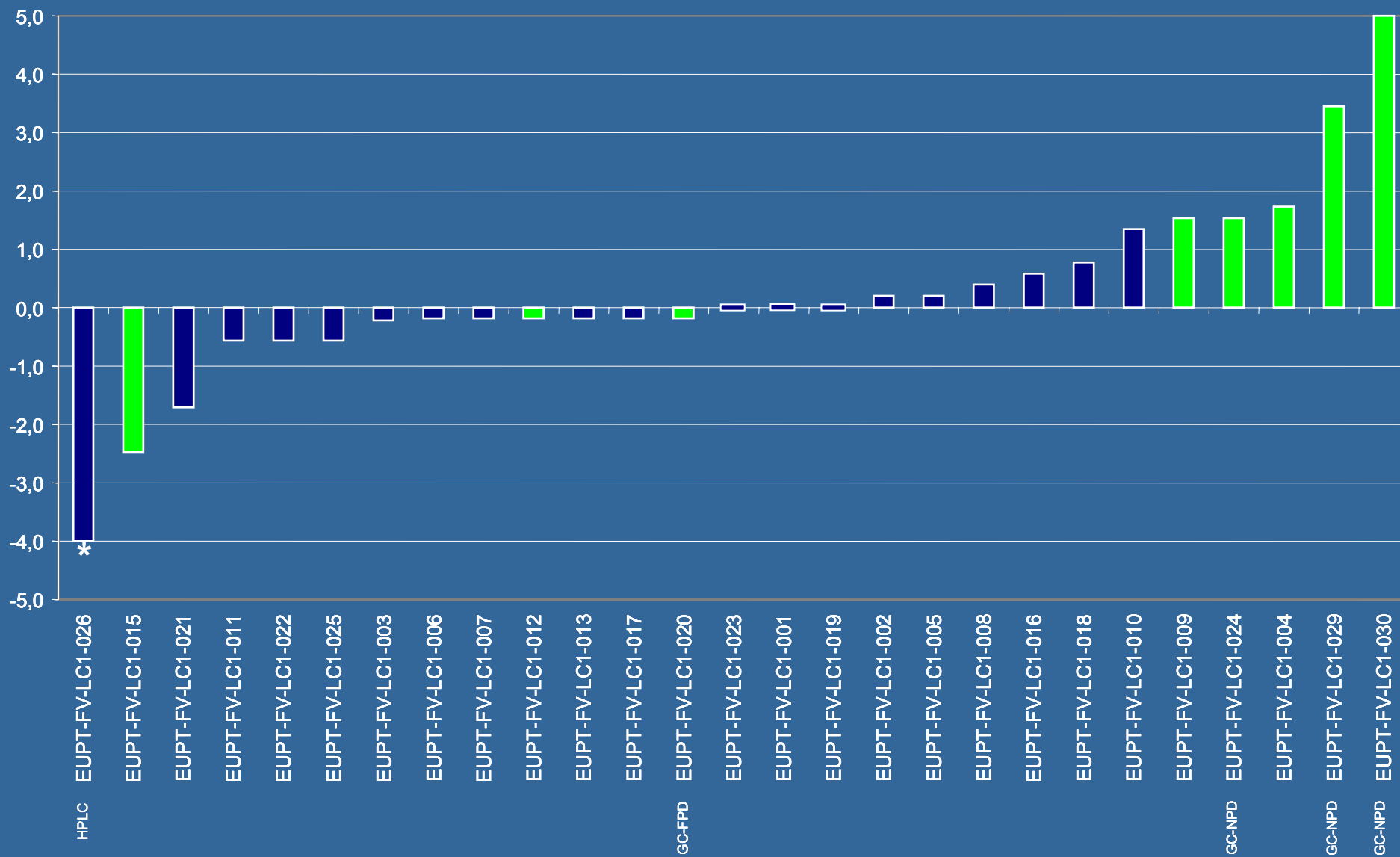
GC * False Negatives
LC
GC and LC

Imidacloprid



- GC
 - LC
 - GC and LC
- * False Negatives

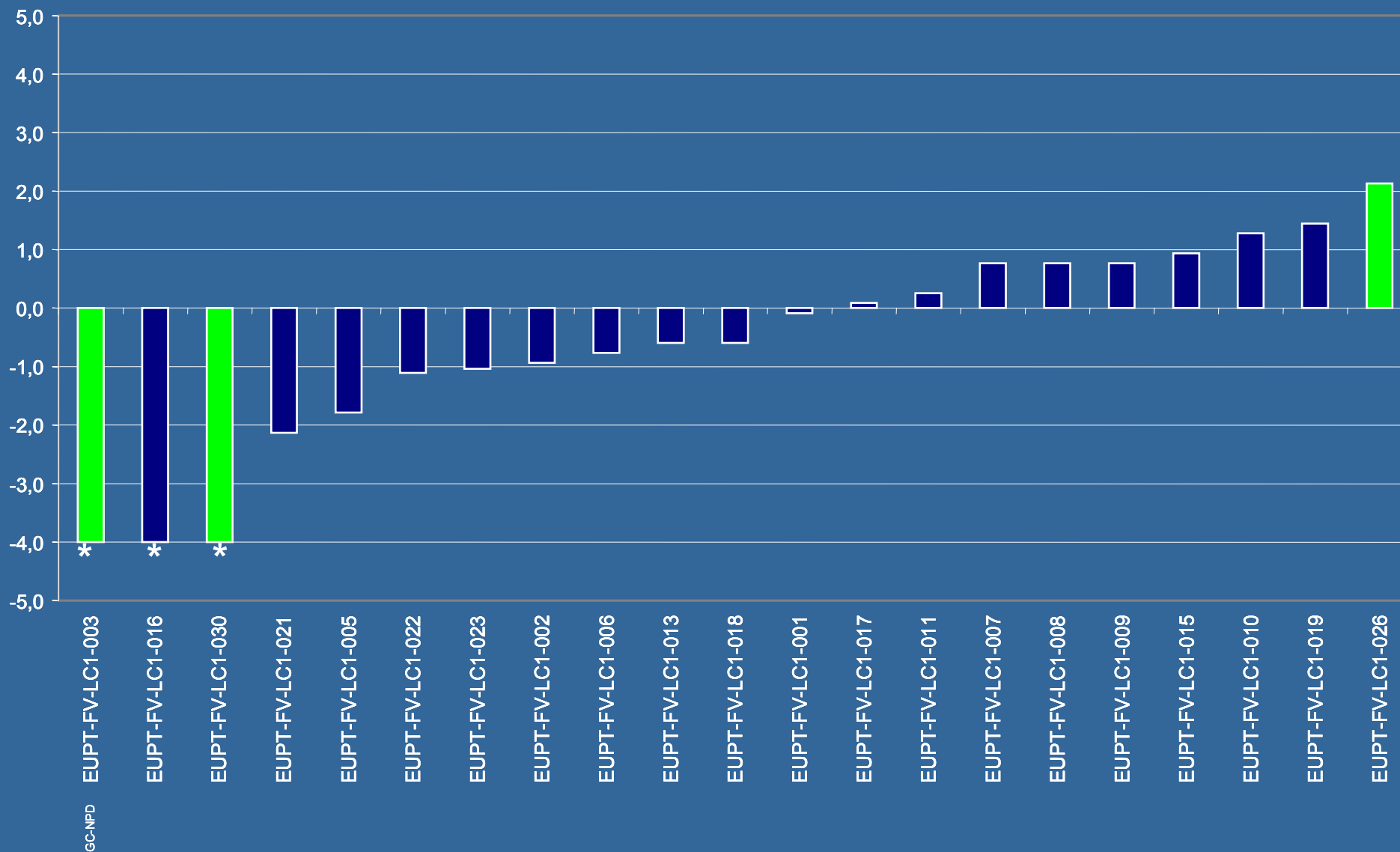
Omethoate



GC
LC
GC and LC

* False Negatives

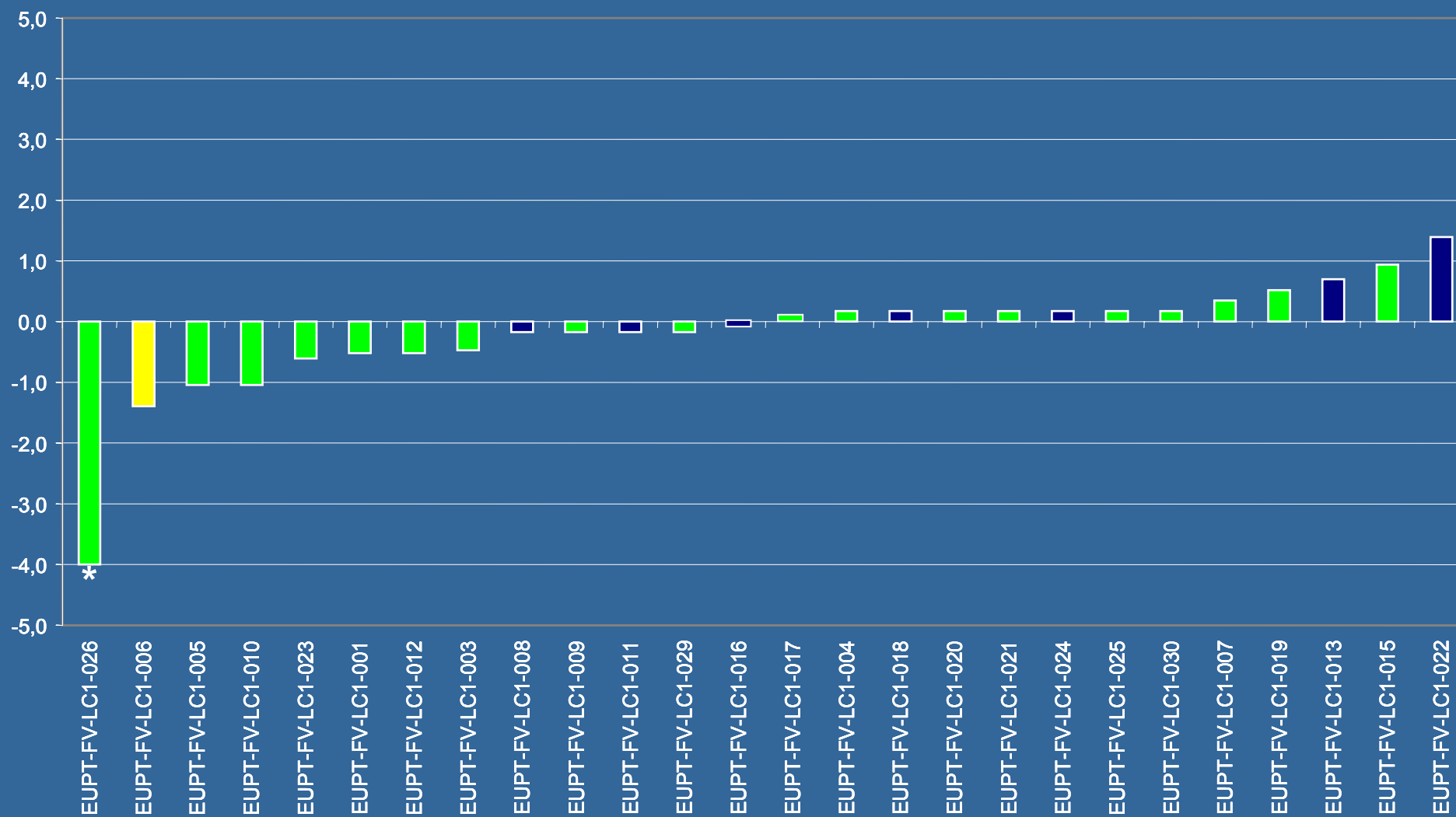
Oxydemeton methyl



GC
LC
GC and LC

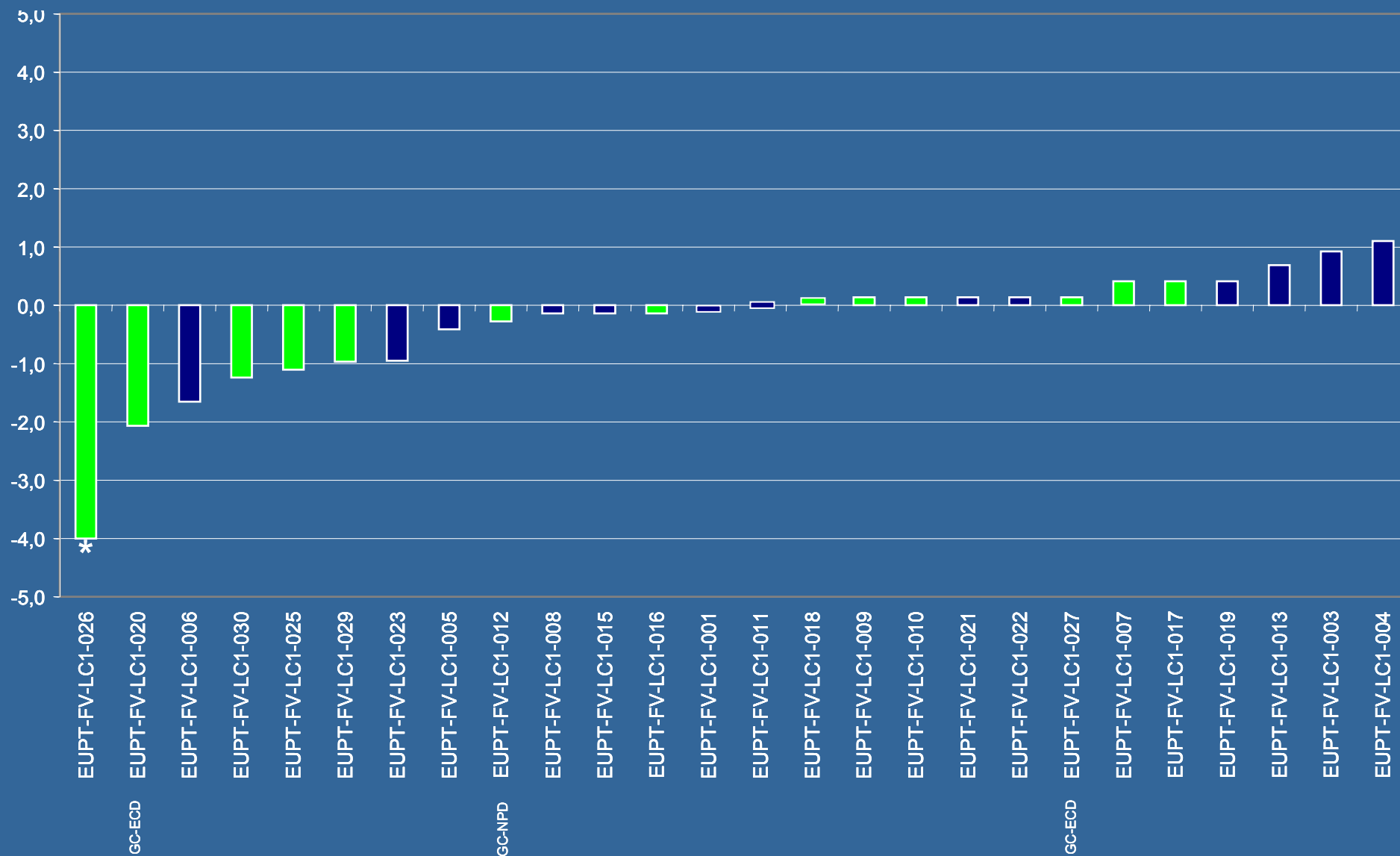
* False Negatives

Pyrimethanil



GC * False Negatives
LC
GC and LC

Tetraconazole



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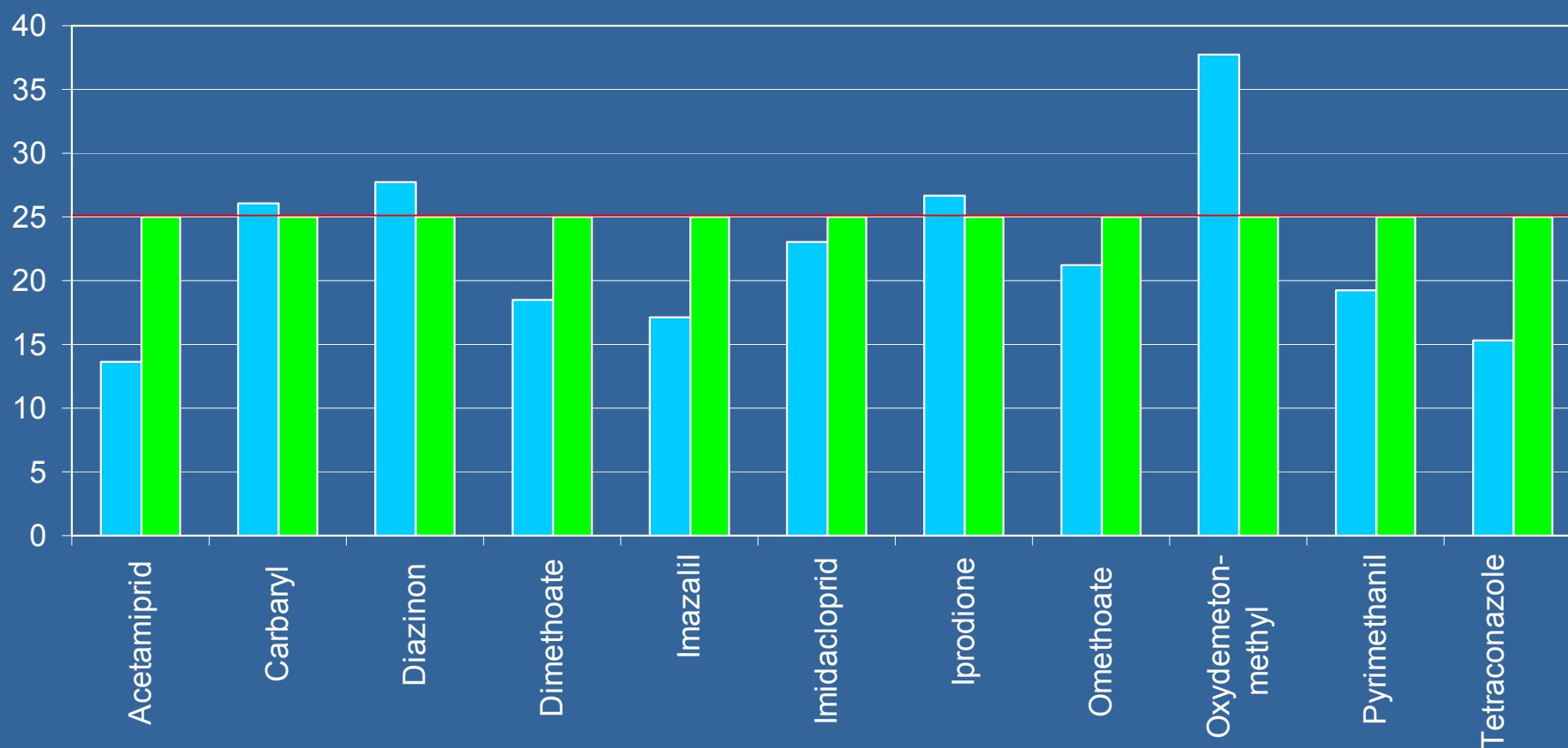
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EUPT-FV-LC1 RSDs

■ Qn RSD
■ FFP RSD



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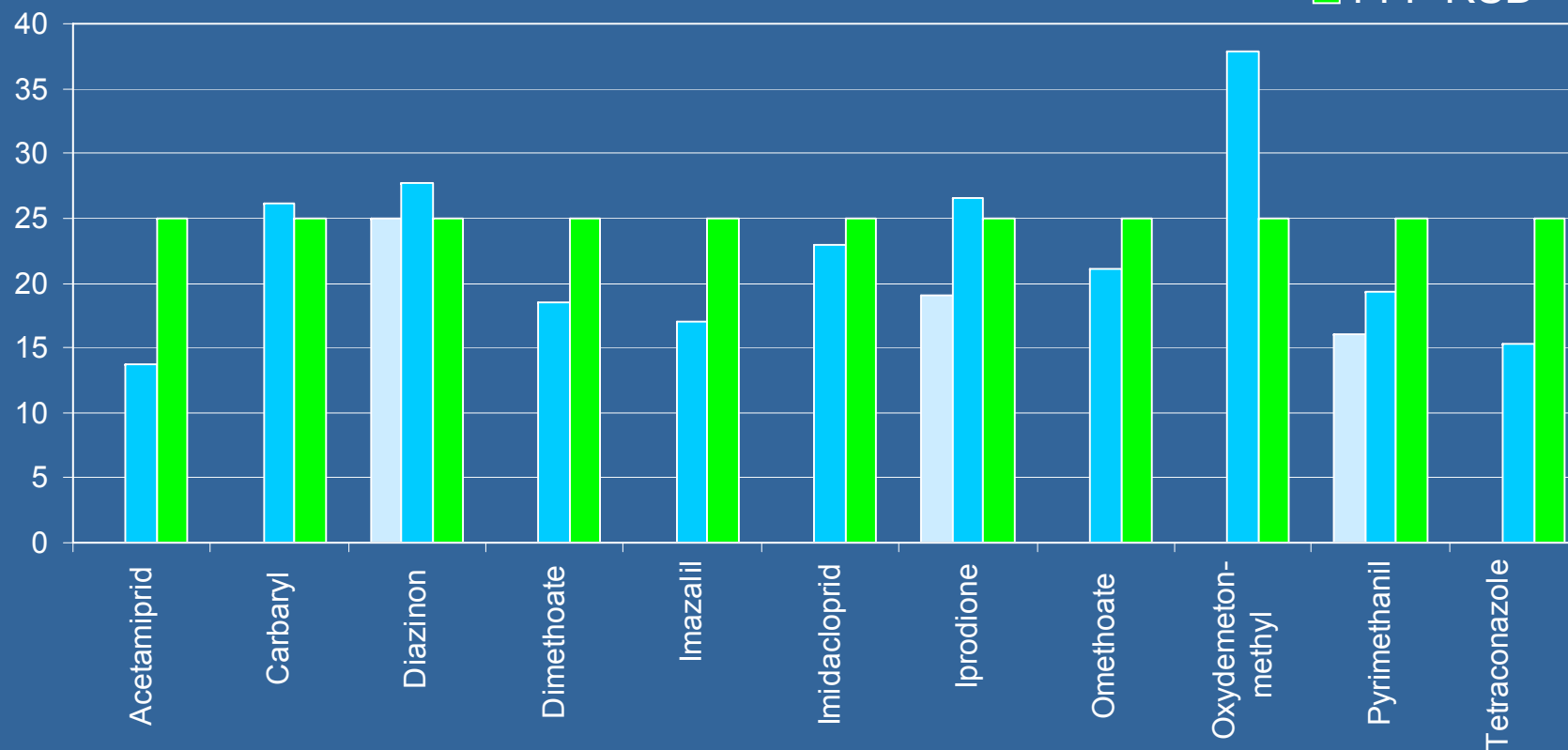
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EUPT-FV9 and LC1 RSDs

■ Qn RSD FV9
■ Qn RSD LC1
■ FFP RSD



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Combined Z-score



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Combined Weight Sum z-Scores

$$WSZ = \frac{\sum_i |z_{\text{accep.}}| \times 1 + \sum_i |z_{\text{quest}}| \times 3 + \sum_i |z_{\text{unaccep.}}| \times 5}{n}$$

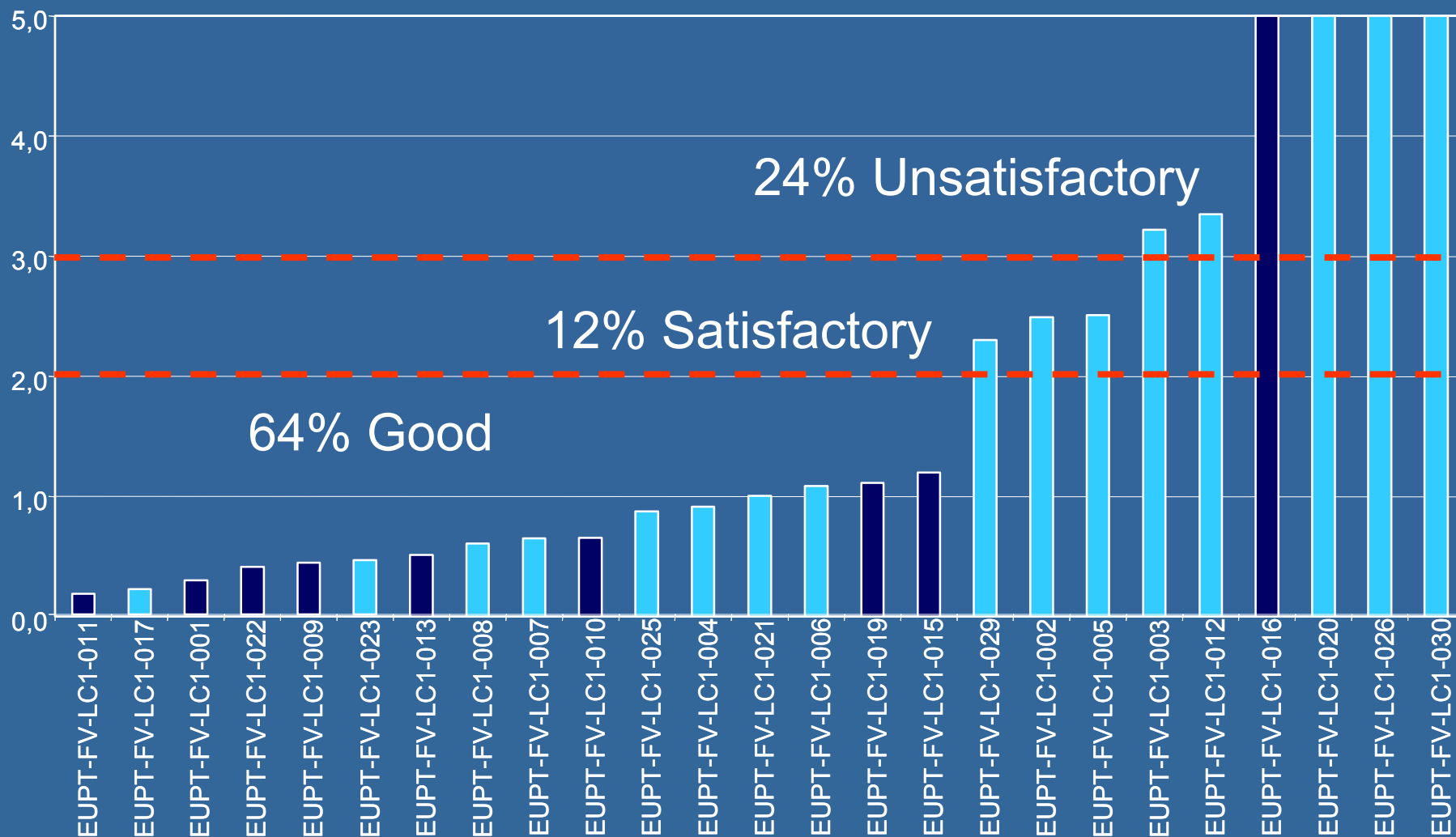
$ z \leq 2$	Good
$2 < z \leq 3$	Satisfactory
$ z > 3$	Unsatisfactory



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EUPF-FV-LC1 WSZ



83% in Category A (25 laboratories from 30)

16 NRLs in Category A 3 in Category B

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Comparative evaluation

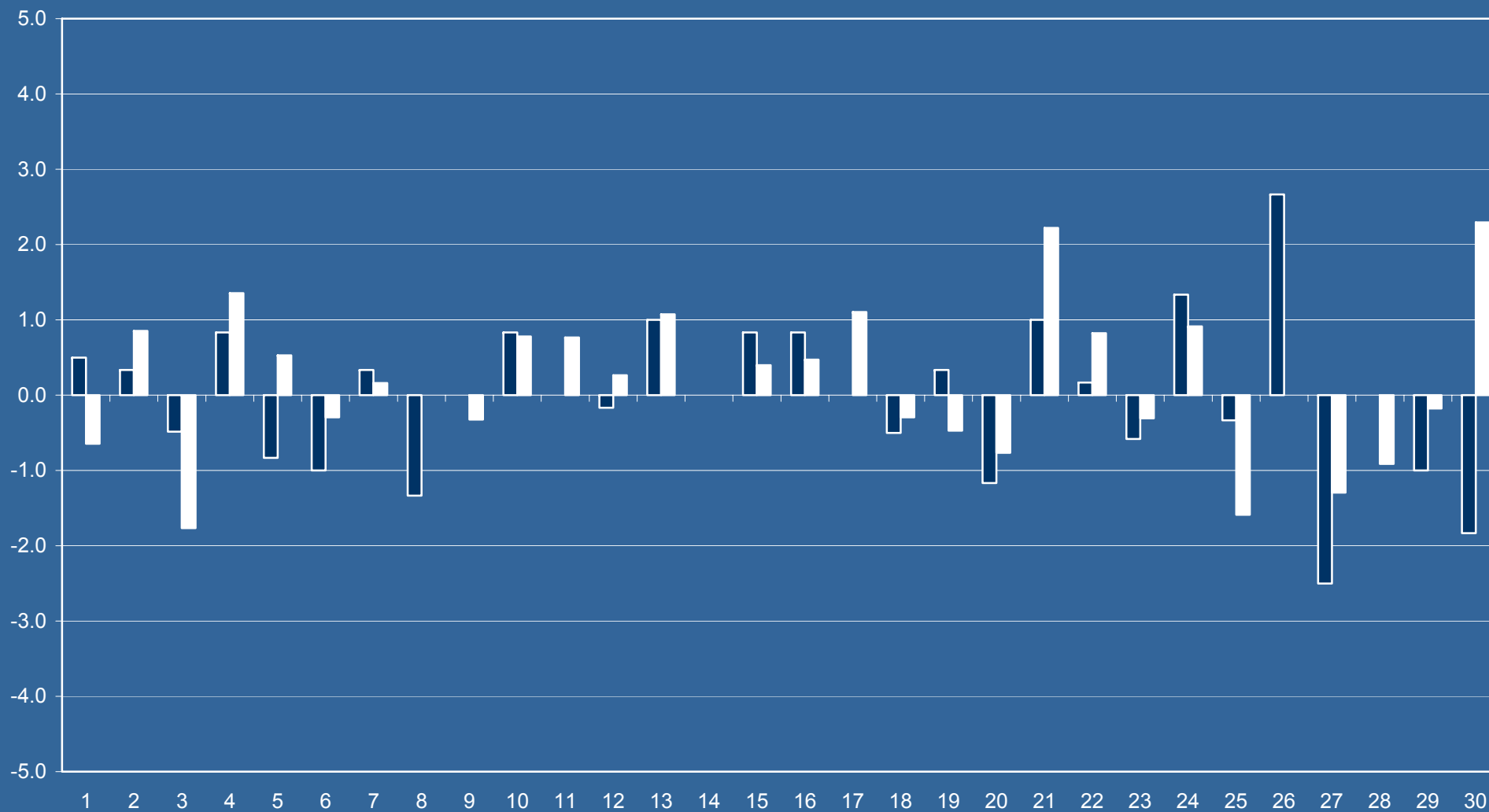


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Diazinon

■ EUPT-FV-LC1
■ EUPT-FV9

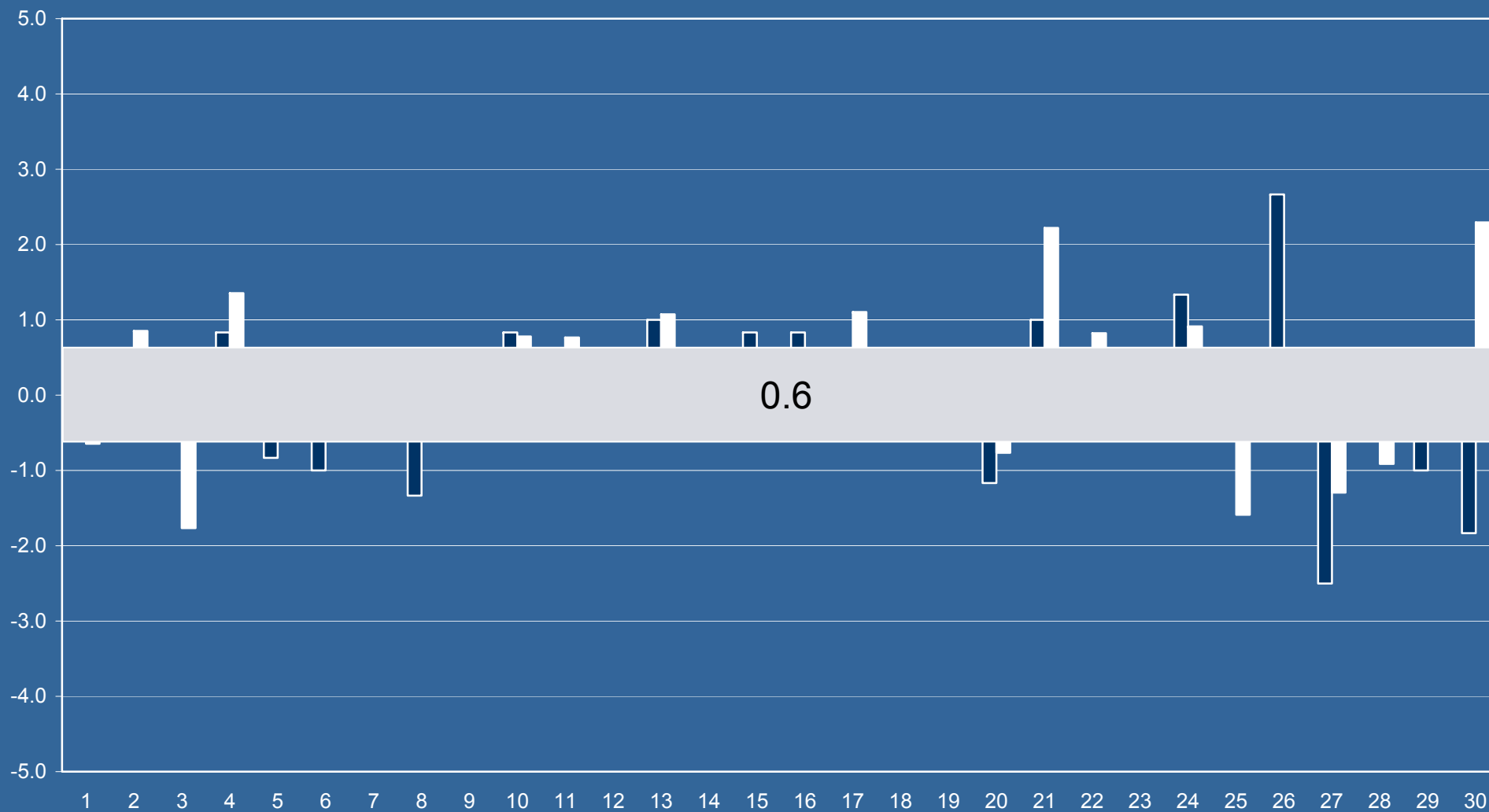


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Diazinon

■ EUPT-FV-LC1
■ EUPT-FV9



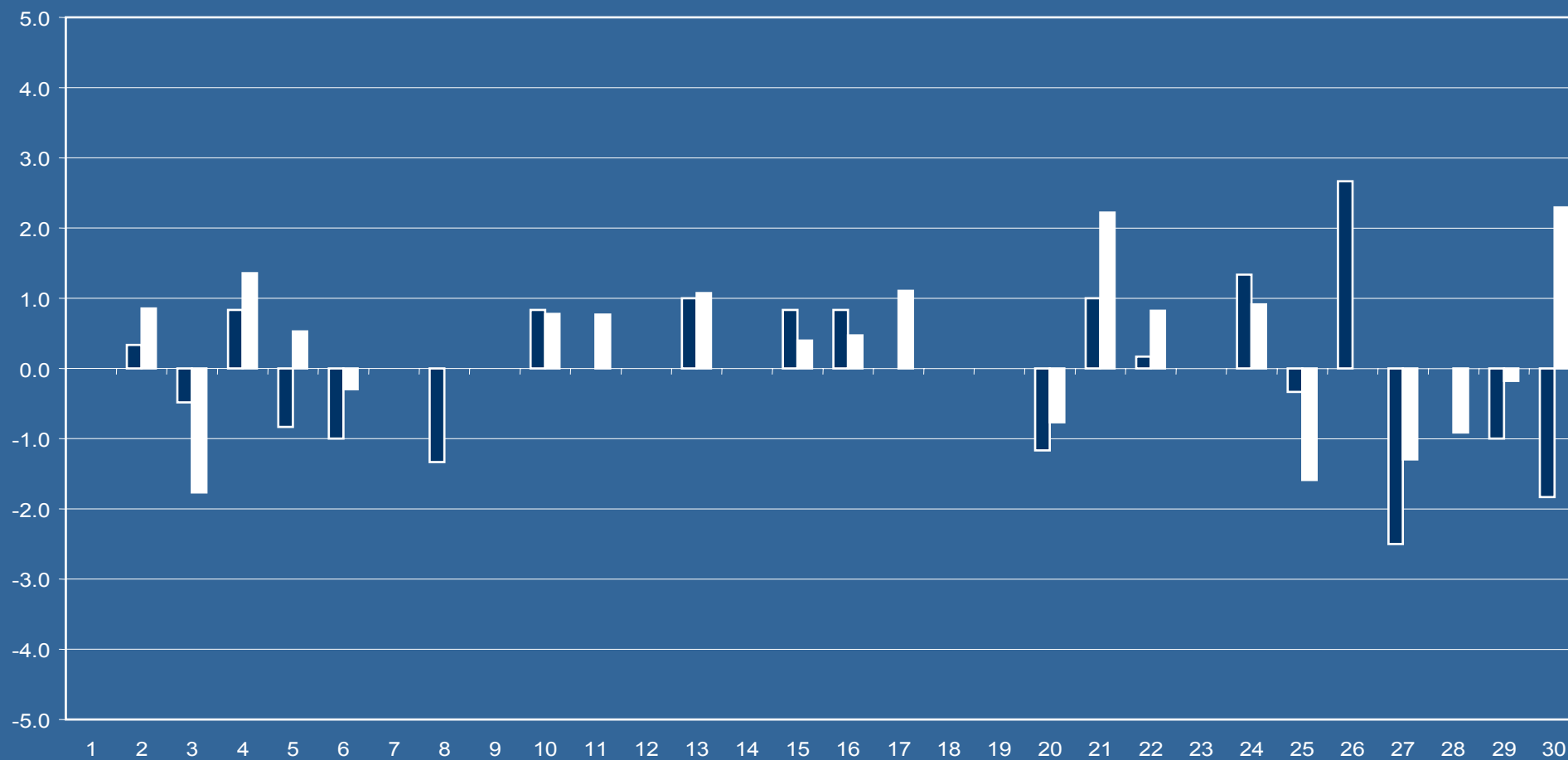
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Diazinon

■ EUPT-FV-LC1
■ EUPT-FV9



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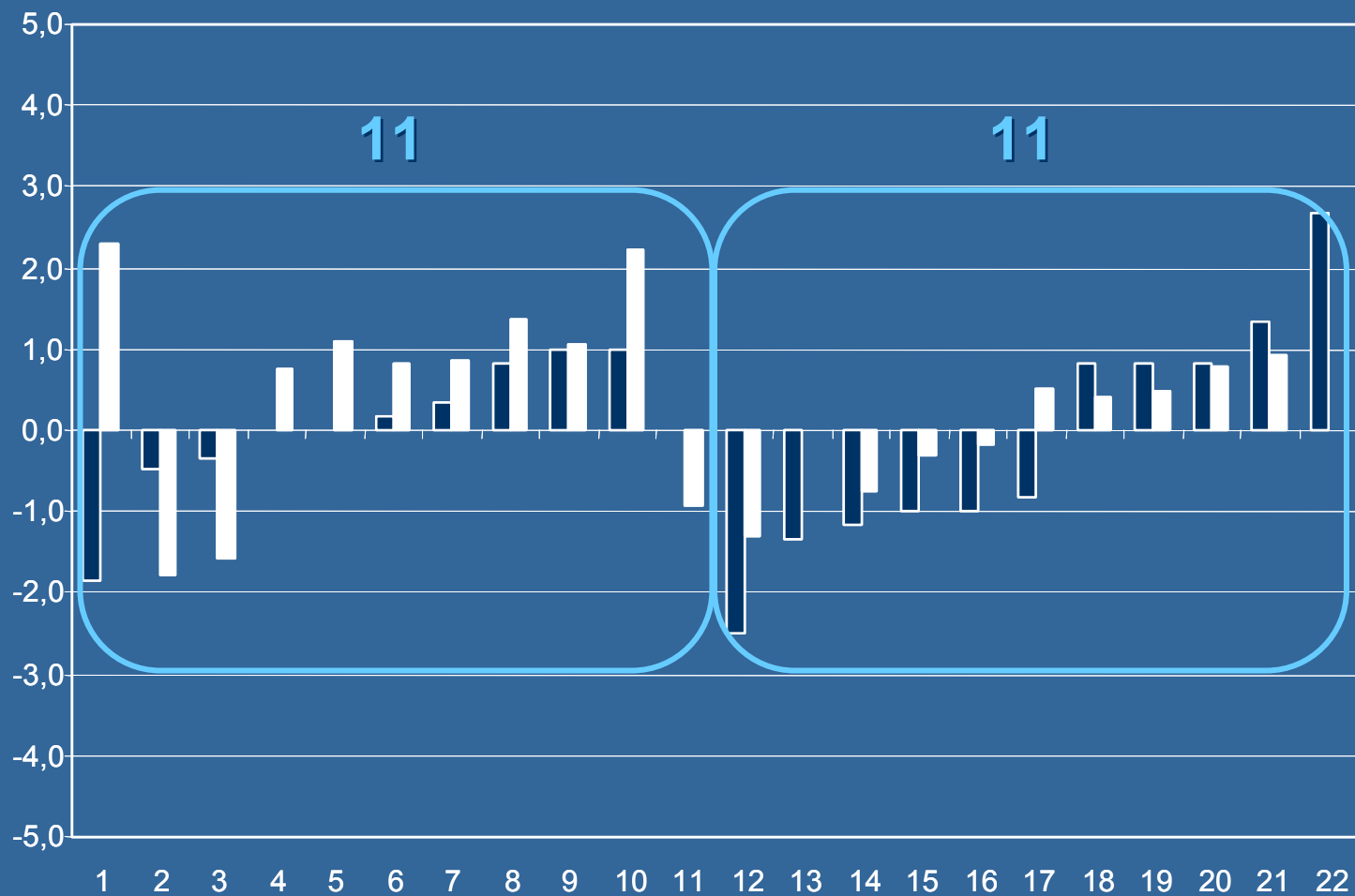
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Valen

Diazinon

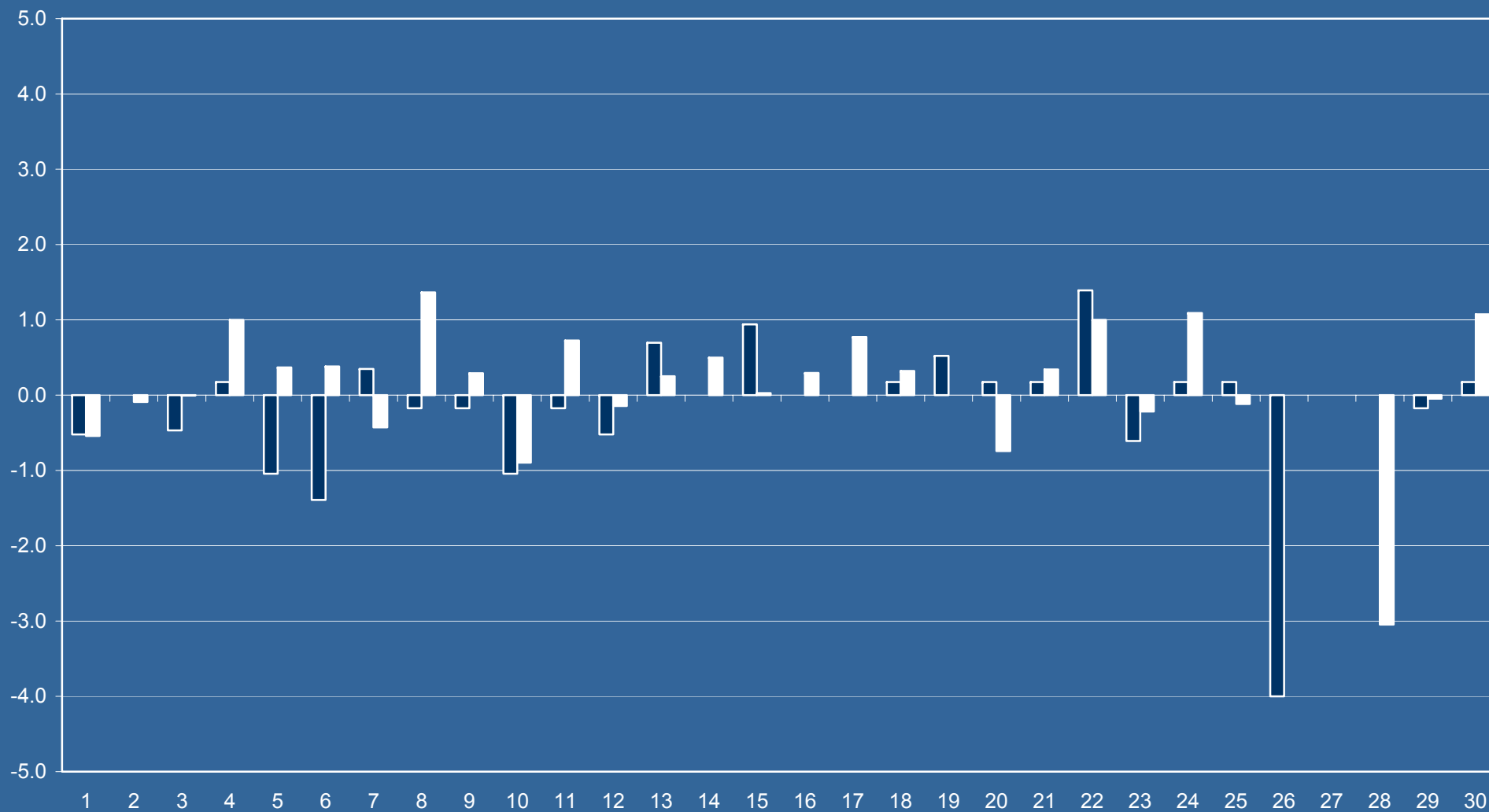
□ EUPT-FV-LC1
■ EUPT-FV9



2007

Pyrimethanil

■ EUPT-FV-LC1
■ EUPT-FV9

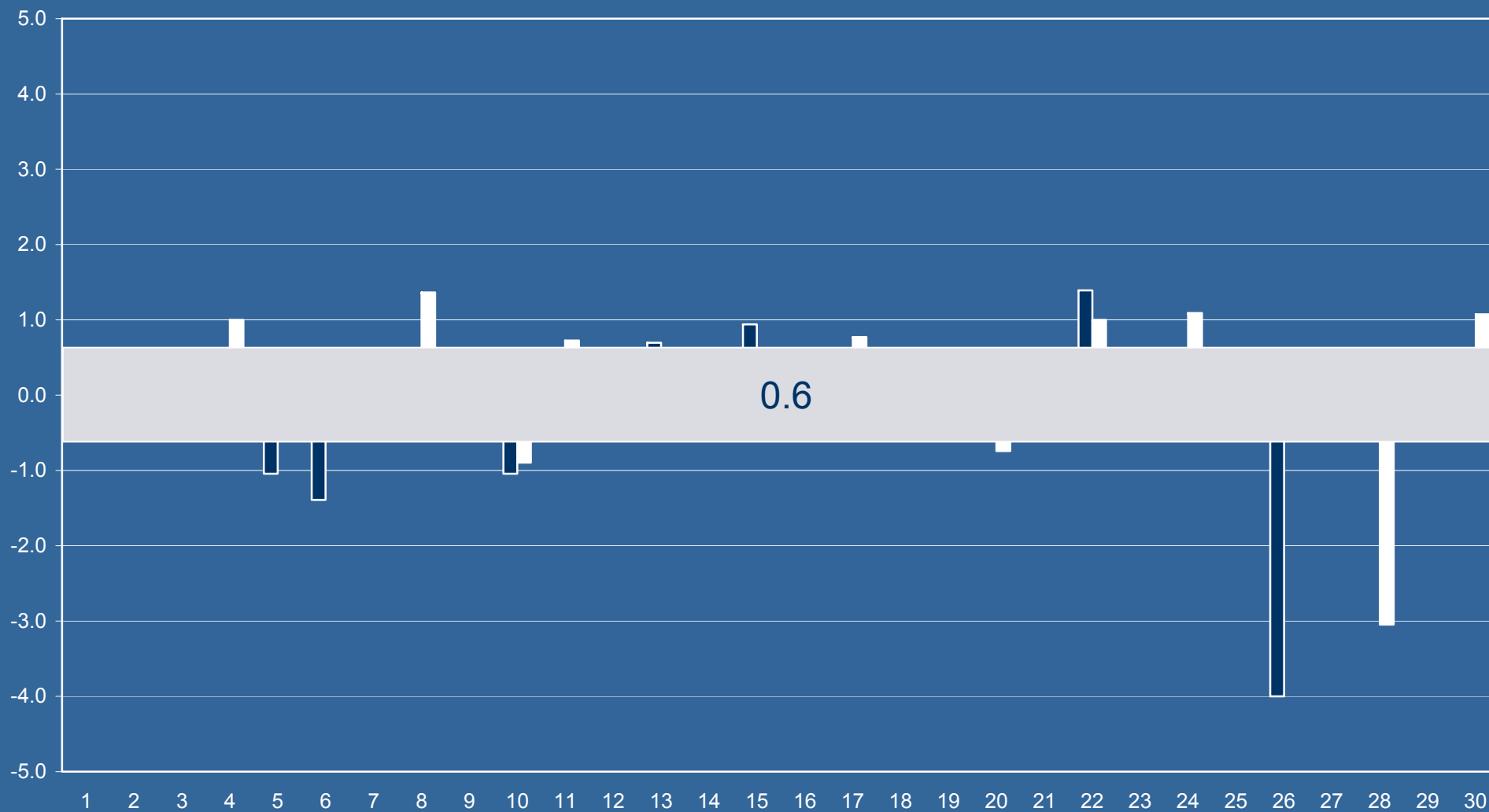


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Pyrimethanil

■ EUPT-FV-LC1
■ EUPT-FV9

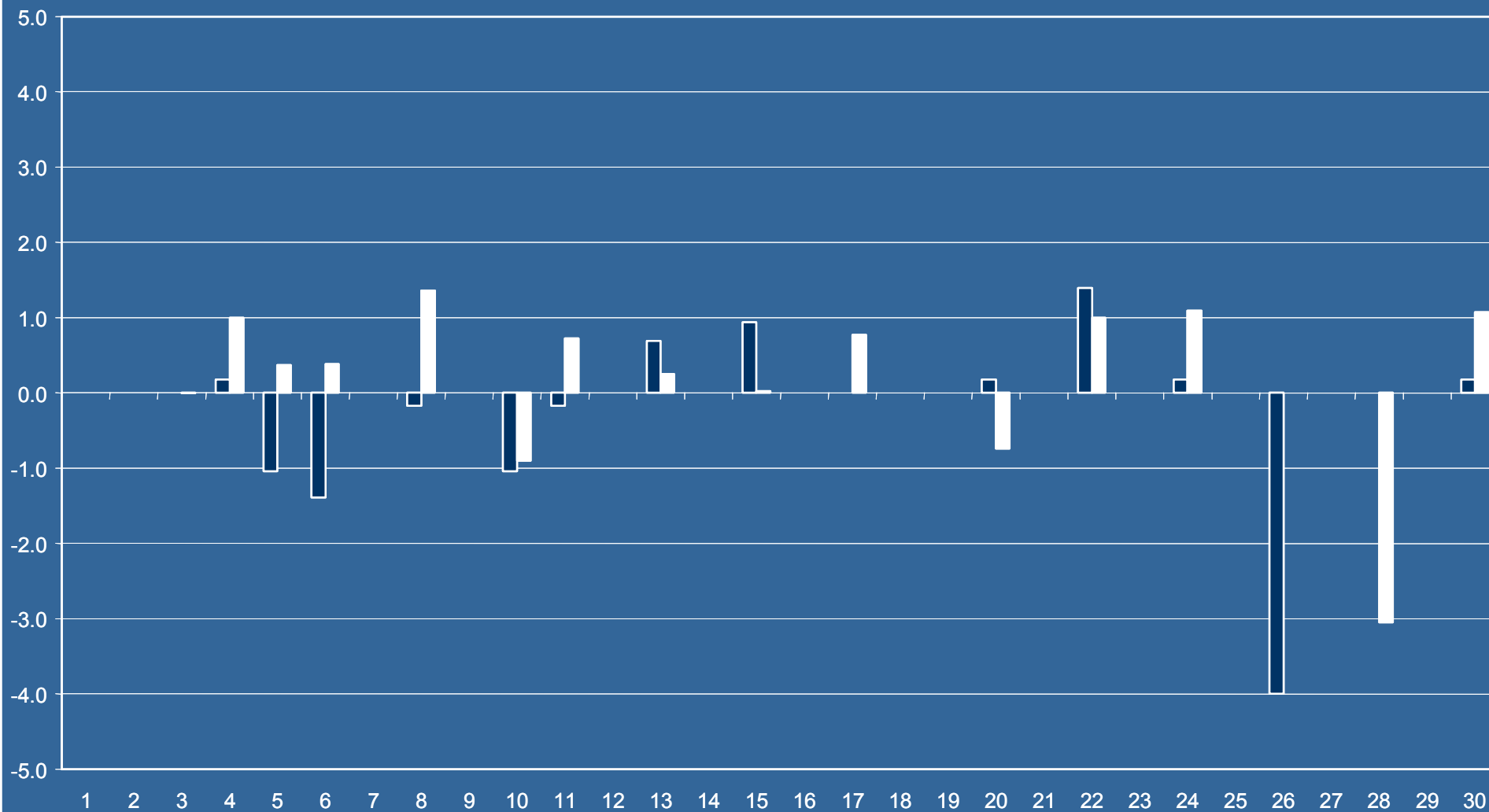


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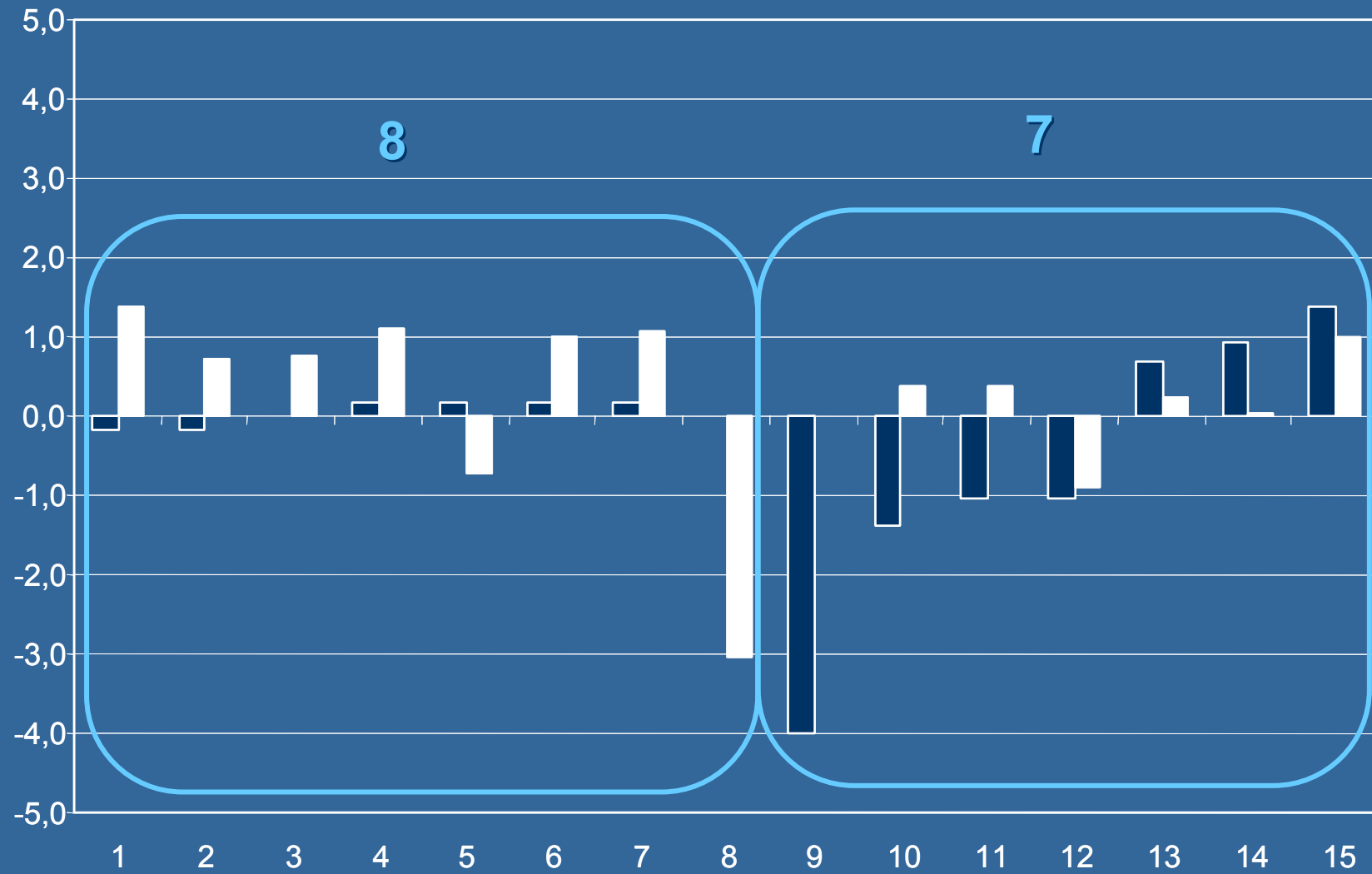
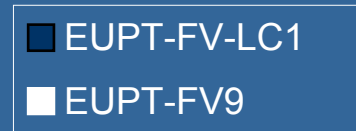
Pyrimethanil

■ EUPT-FV-LC1
■ EUPT-FV9



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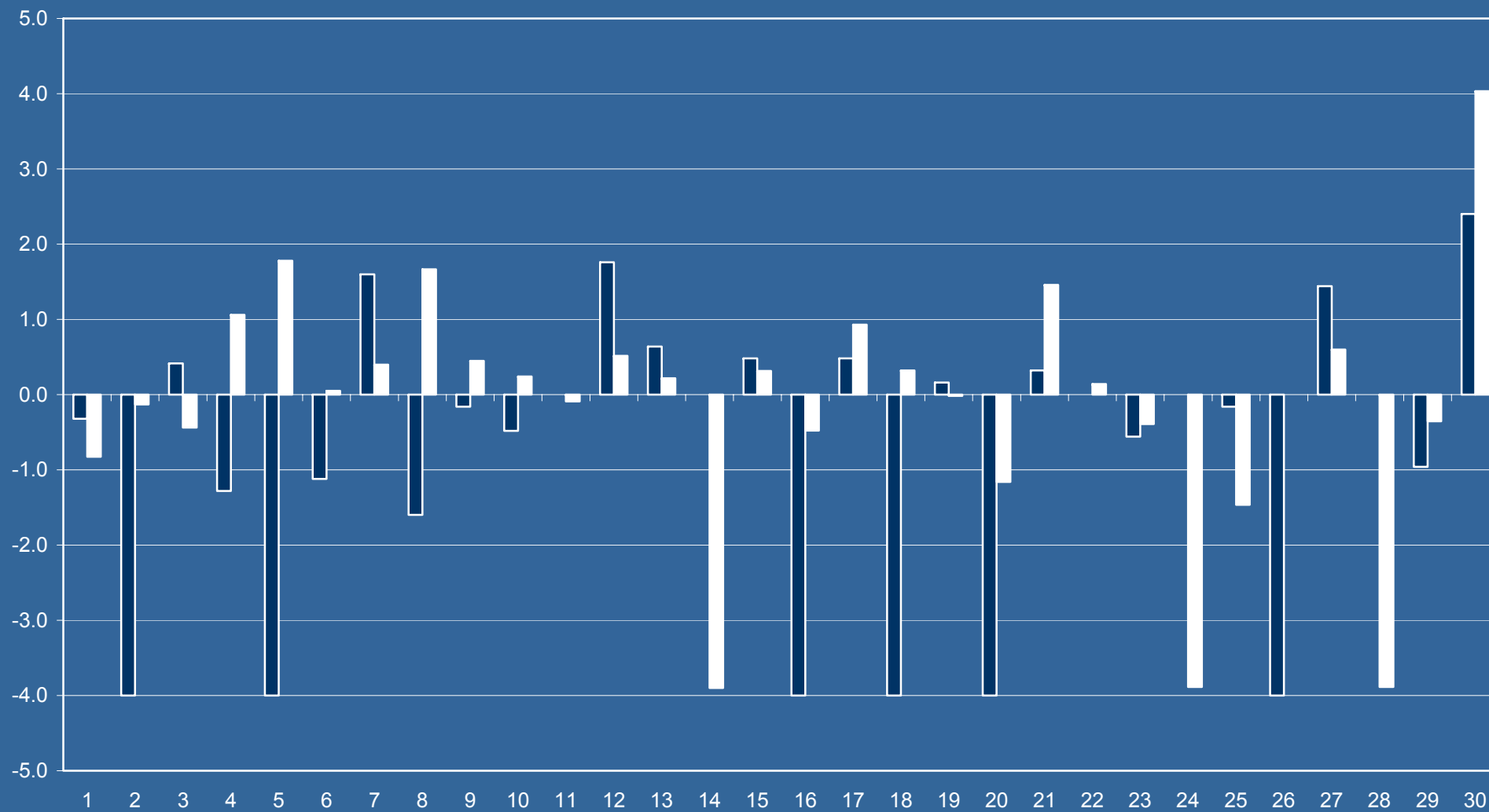
Pyrimethanil



2007

Iprodione

■ EUPT-FV-LC1
■ EUPT-FV9

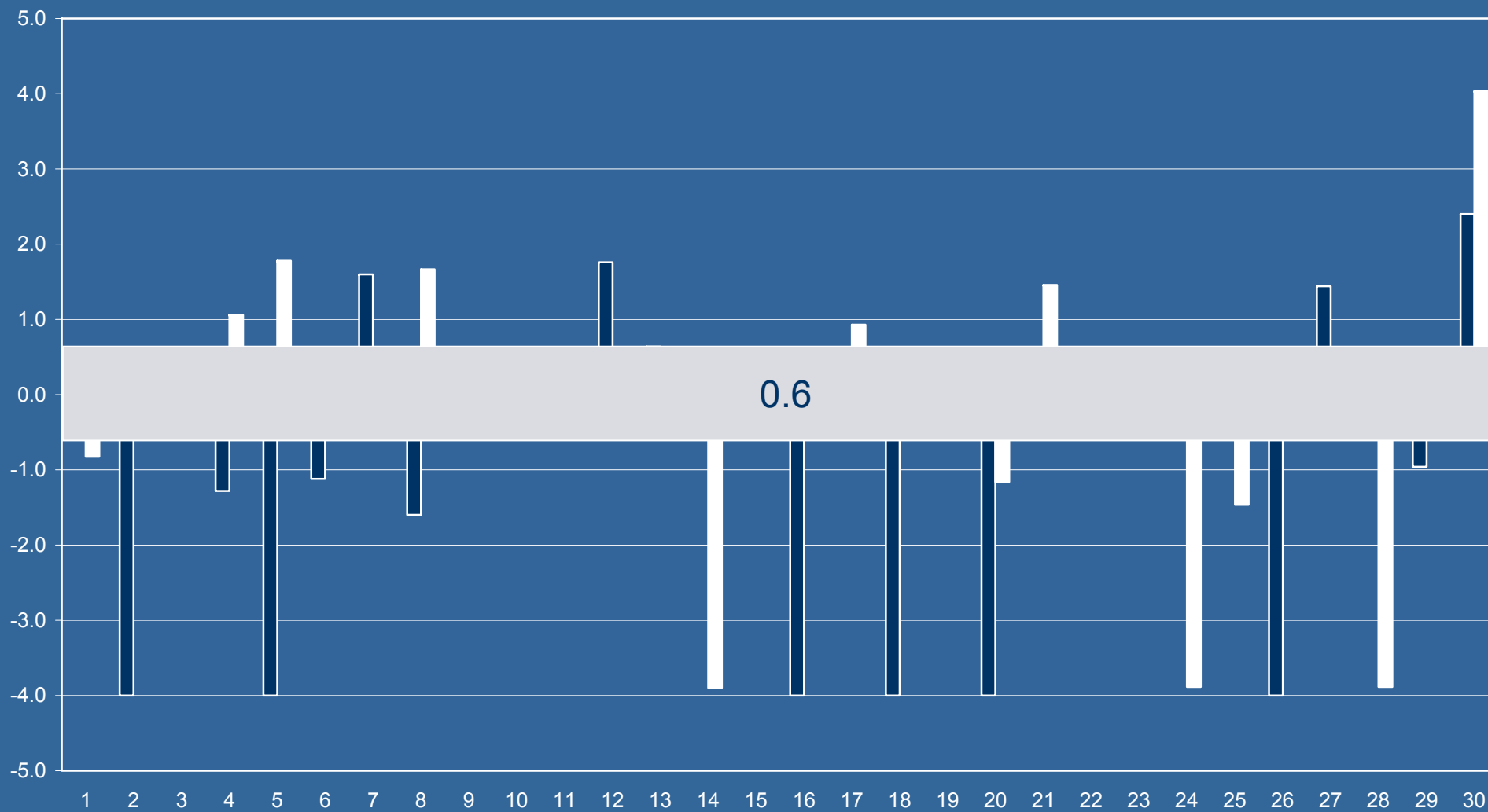


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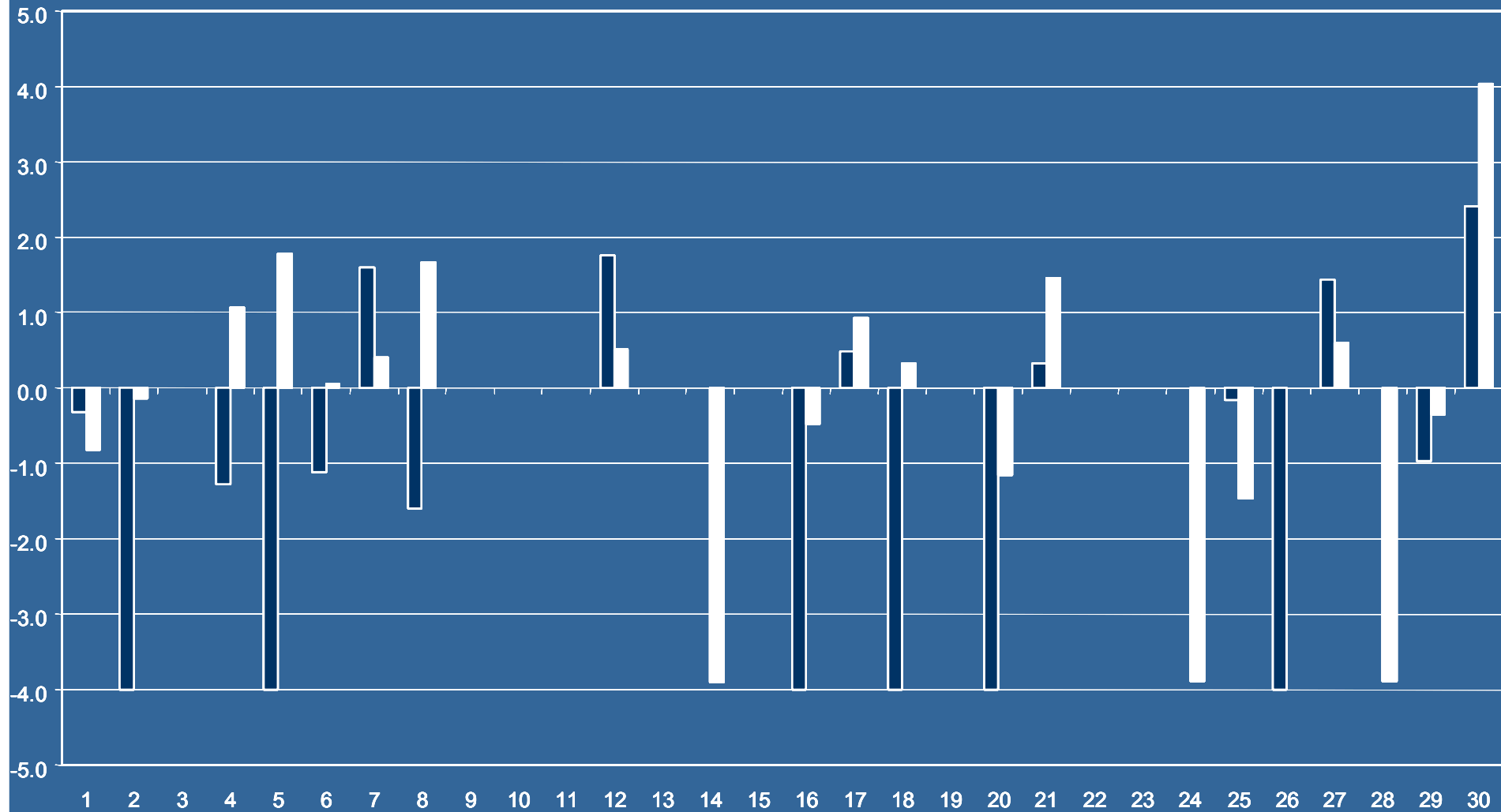
■ EUPT-FV-LC1
■ EUPT-FV9



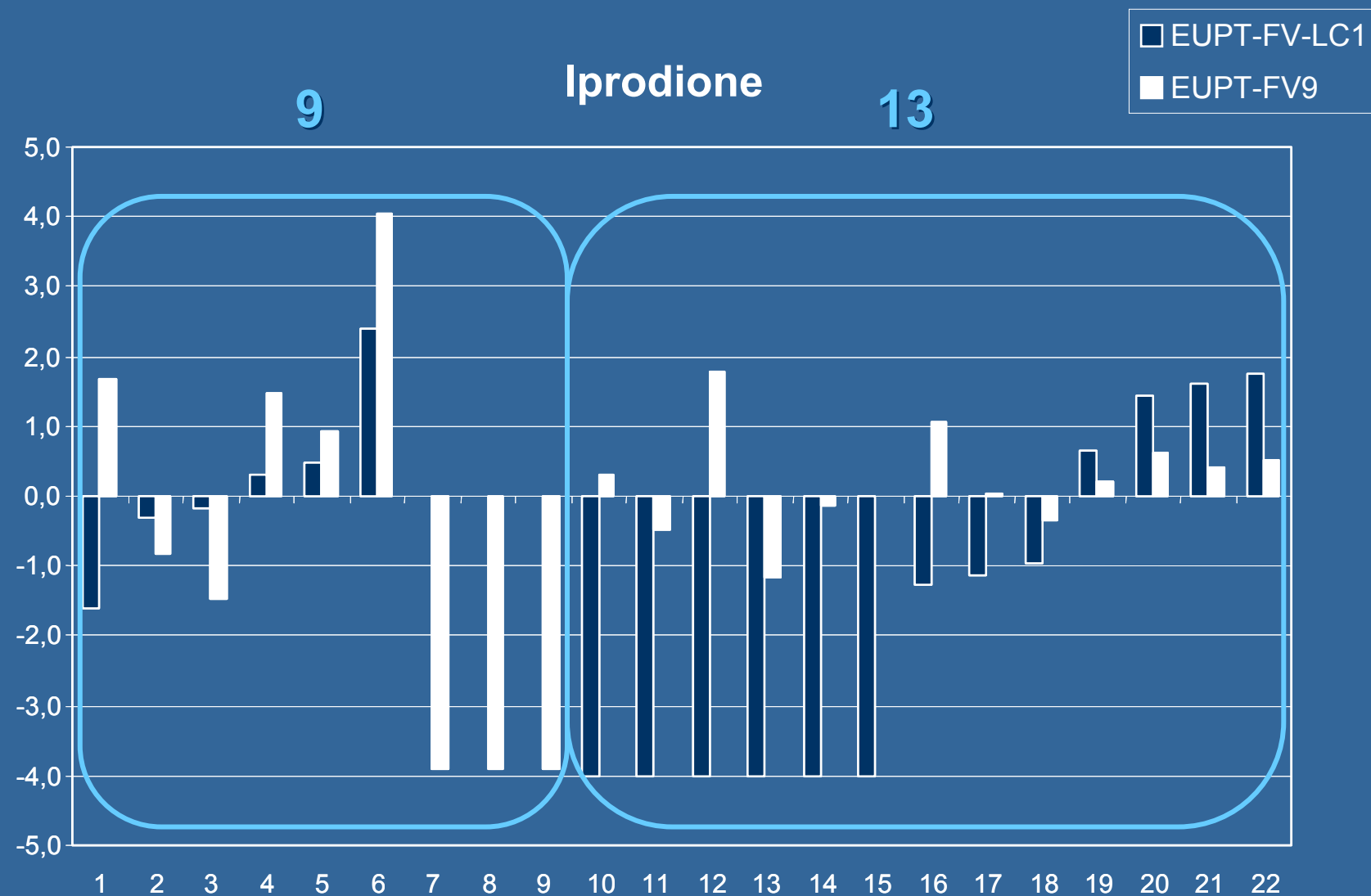
0.6

Iprodione

■ EUPT-FV-LC1
■ EUPT-FV9



Iprodione



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PESTICIDE RESIDUES IN FOOD

26-29 September 2007
Valencia – SPAIN

ANOVA Test

P-Value between groups

Diazinon	0.268
Pyrimethanyl	0.362
Iprodione	0.209

P Target Value: 0.05



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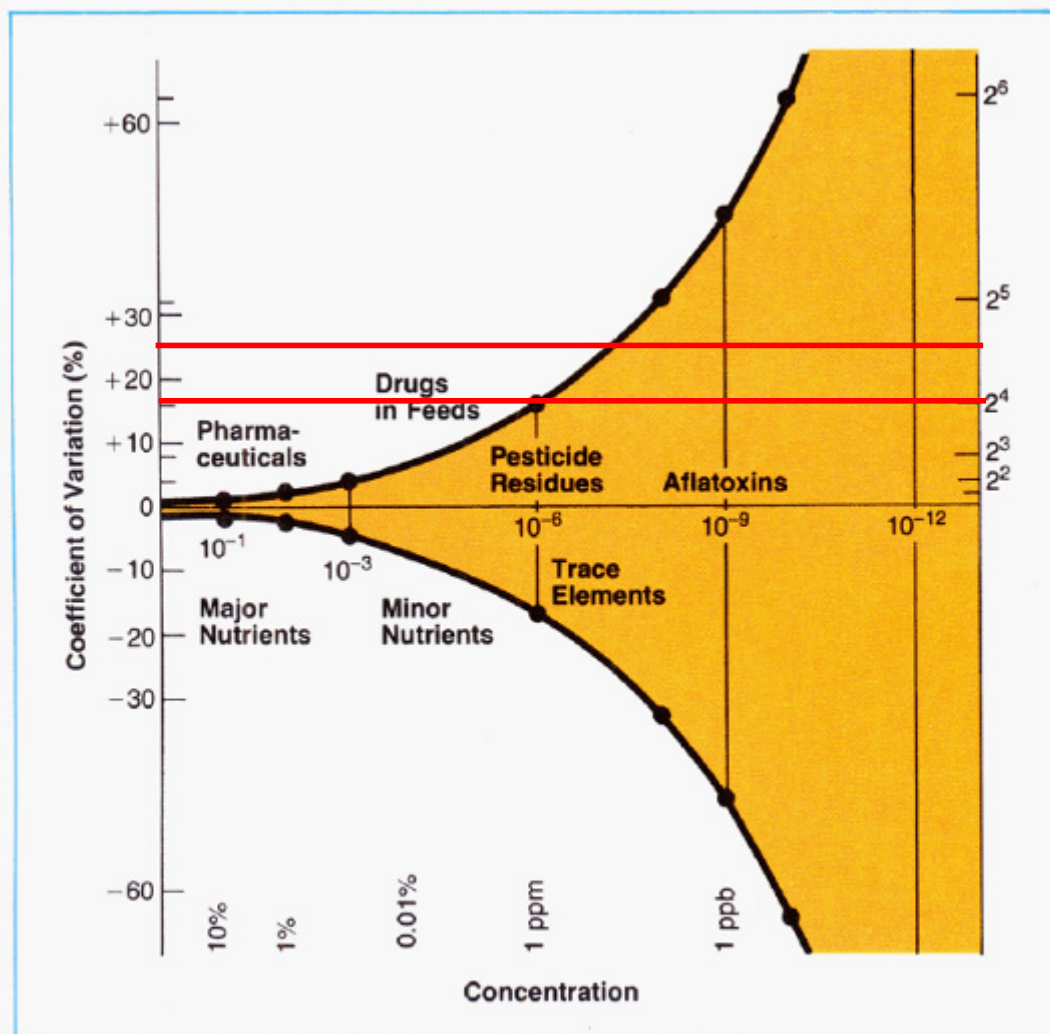


Figure 1. The general curve relating interlaboratory coefficients of variation (expressed as powers of two on the right) with concentration (expressed as powers of 10) along the horizontal center axis

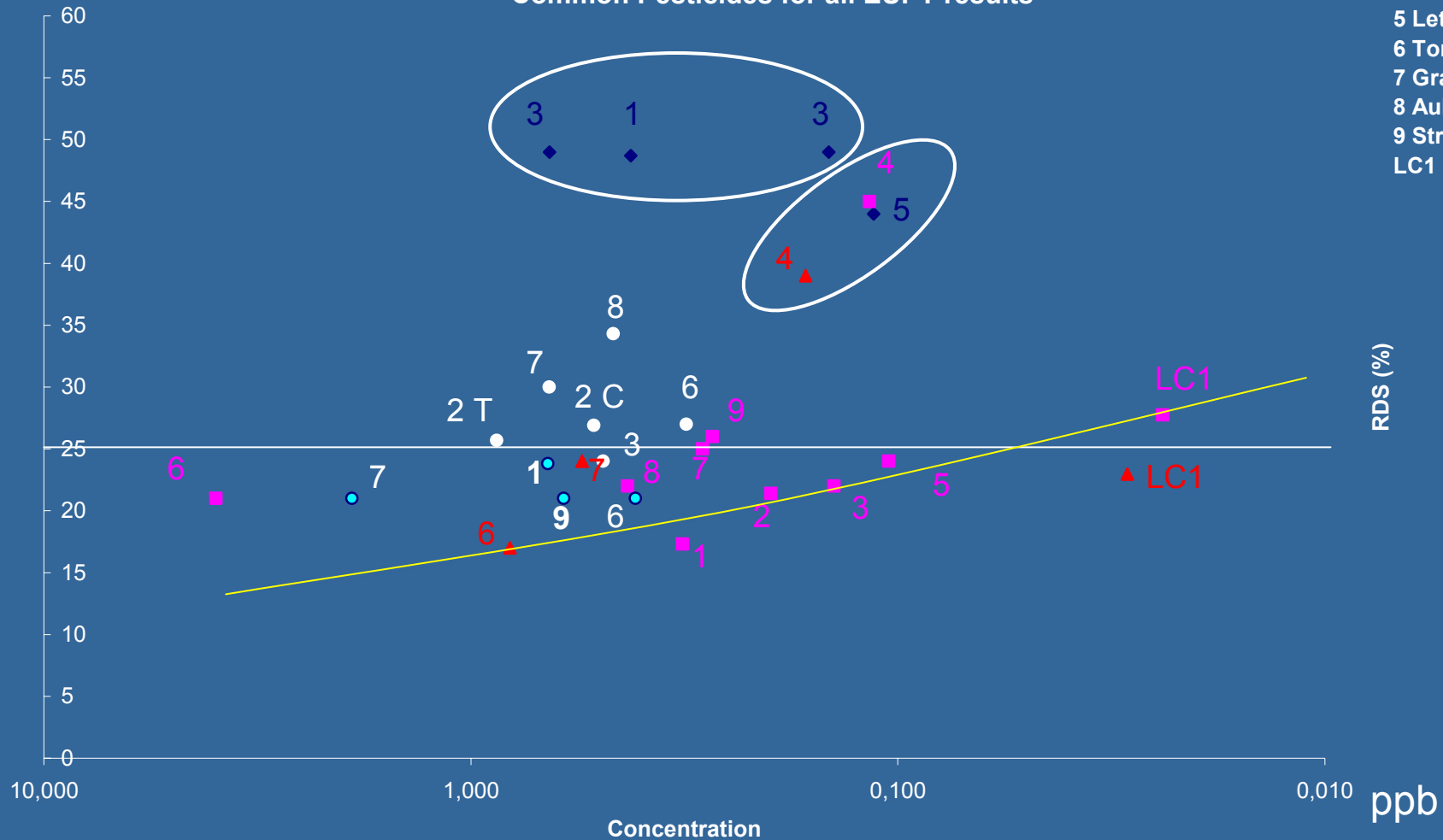


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RDS(%)

Common Pesticides for all EUPT results

- 1 Peppers
- 2 Apples
- 3 Cucumber
- 4 Oranges
- 5 Lettuce
- 6 Tomatoes
- 7 Grapes
- 8 Aubergine
- 9 Strawberry
- LC1 Pears



◆ Acephate/Methamidophos

■ Diazinon

▲ Imidacloprid

● Procydimone

● Carbendazim/Thiabendazole

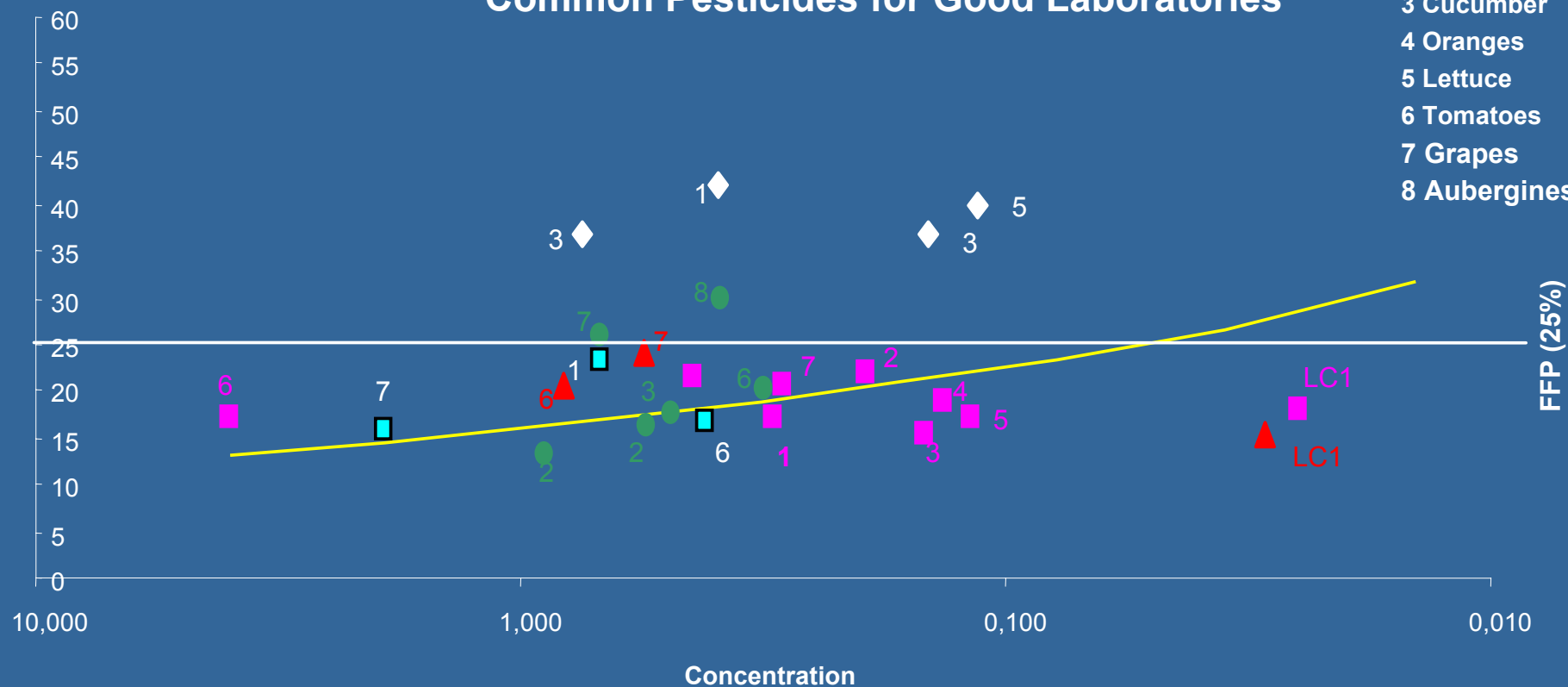
— Horwitz

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Common Pesticides for Good Laboratories

- 1 Peppers
- 2 Apples
- 3 Cucumber
- 4 Oranges
- 5 Lettuce
- 6 Tomatoes
- 7 Grapes
- 8 Aubergines

Qn RSD(%)



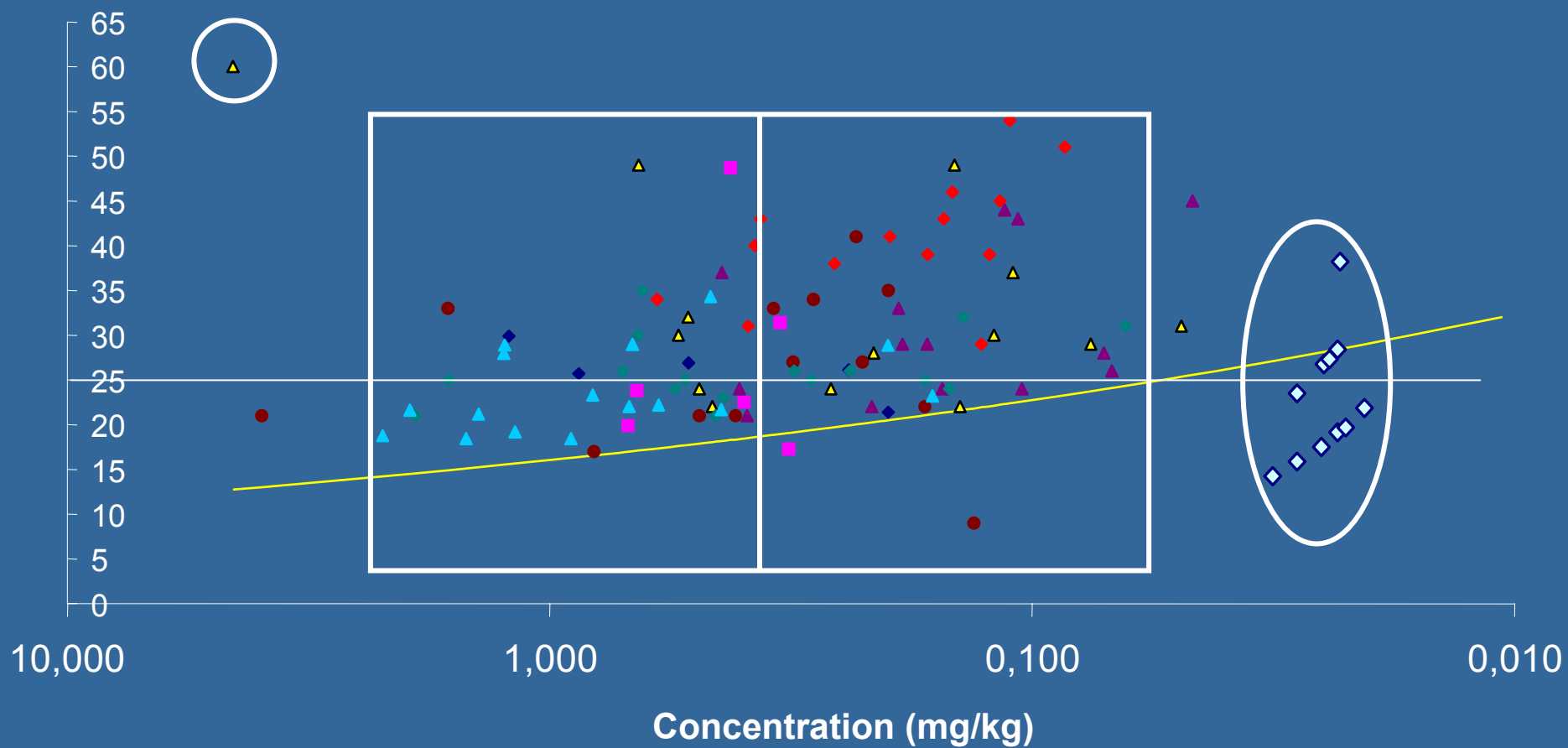
◆ Acephate/Methamidophos ■ Diazinon ▲ Imidacloprid ■ Procymidone ● Carbendazim/Thiabendazole — Horwitz

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Qn RSD (%)

AII EUPT



■ PT1 ◆ PT2 ▲ PT3 ◆ PT4 ▲ PT5 ● PT6 ◆ PT7 ▲ PT8 ◆ LC1 — Horwitz

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CONCLUSIONS



No relevant differences are obtained in the evaluation parameters and results for the EUPT LC respect to the “regular” concentration PTs. But, more data /information is required

Only the number of false negatives at LC could represent an important point to be considered.

The homogenization procedure applied fit all the requirements for PTs.

In the future a combination of pesticides at “regular” and low concentration on the same PT sample is possible and can facilitate a more adequate and realistic evaluation of the performance.



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THANK YOU FOR YOUR ATTENTION



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EUPT-FV-LC1 z-scores

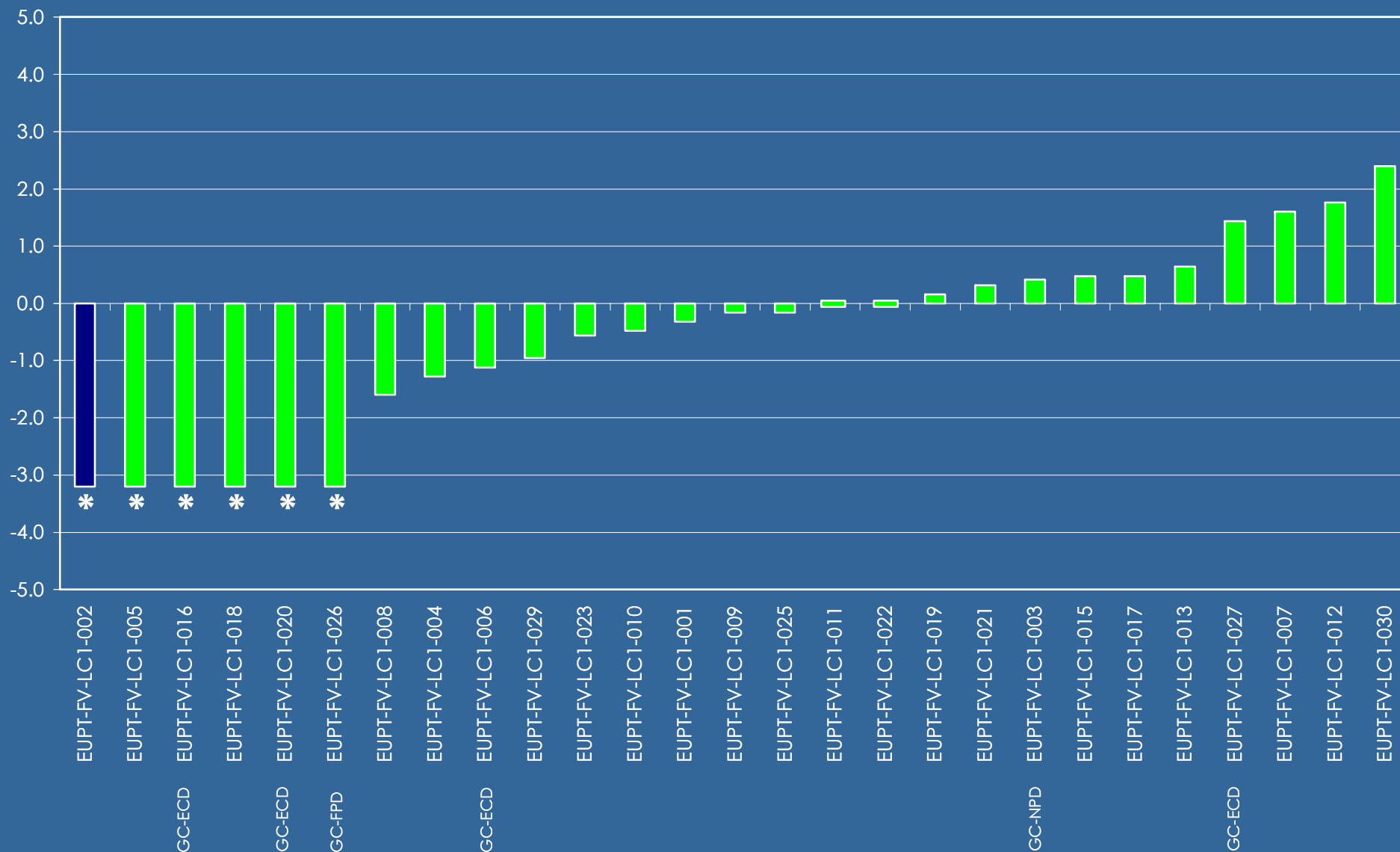


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- GC
- LC
- GC and LC

* False Negatives

Iprodione



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PESTICIDES	Median (mg/Kg)	Nº of reported results	Nº of NA (not sought)	False negatives	% of results from the total 30
Acetamiprid	0.033	26	1	1	87
Carbaryl	0.026	26	2	0	87
Diazinon	0.024	28	0	0	93
Dimethoate	0.024	27	0	1	90
Imazalil	0.026	22	3	3	73
Imidacloprid	0.029	24	2	2	80
Iprodione	0.025	22	1	5	73
Omethoate	0.021	26	1	1	87
Oxydemeton-methyl	0.024	18	7	3	60
Pyrimethanil	0.023	25	2	1	83
Tetraconazole	0.029	25	2	1	83

Total

269

18 (6.7%)



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26-29 September 2007
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EU PT08

PESTICIDES	Nº of reported results	Nº of NA (not sought)	False negatives	% of results from the total 128
Acetamiprid	78	47	3	61
Azoxystrobin	113	15	0	88
Bifenthrin	119	9	0	93
Bromopropylate	125	3	0	98
Carbaryl	108	17	3	84
Carbendazim	94	34	0	74
Chlorpyrifos	127	1	0	99
Cyprodinil	114	13	1	89
Diazinon	127	1	0	99
Dichlofluanid	113	5	10	88
Fludioxonil	92	33	3	72
Imazalil	107	14	7	84
Lambda-cyhalothrin	121	5	2	95
Myclobutanil	114	14	0	89
Parathion	114	10	4	89
Pirimicarb	114	14	0	89

Total

1824

31 (1.7%)

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[illegible]

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PESTICIDE RESIDUES IN FOOD



	Acetamiprid	Carbaryl	Diazinon	Dimethoate	Imazalil	Imidacloprid	Iprodione	Omethoate	Oxydemeton-methyl	Pyrimethanil	Tetraconazole
Day 1 (1 st sample)	0.037	0.023	0.032	0.023	0.029	0.024	0.031	0.029	0.026	0.025	0.035
Day 1 (2 nd sample)	0.033	0.024	0.028	0.024	0.028	0.025	0.027	0.023	0.023	0.024	0.033
Mean 1	0.035	0.023	0.030	0.023	0.028	0.024	0.029	0.026	0.024	0.024	0.034
Day 2 (1 st sample)	0.036	0.024	0.033	0.022	0.027	0.023	0.031	0.028	0.024	0.025	0.034
Day 2 (2 nd sample)	0.033	0.025	0.029	0.024	0.030	0.028	0.026	0.025	0.024	0.023	0.035
Mean 2	0.034	0.024	0.031	0.023	0.028	0.025	0.028	0.026	0.024	0.024	0.034
% Critical	5	5	5	5	5	5	5	5	5	5	5
% real	1.4	4.25	3.3	2.1	1.2	4.1	1.7	1.9	2.0	2.0	1.5

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Countries	No of Labs
AUSTRIA	1
BELGIUM	1
BULGARY	1
CYPRUS	1
CZECH REPUBLIC	1
FINLAND	1
FRANCE	1
GERMANY	9
GREECE	1
HUNGARY	1
IRELAND	1
ITALY	1
LATVIA	1
POLAND	1
PORTUGAL	1
SLOVAKIA	1
SPAIN	2
SWEDEN	2
THE NETHERLANDS	1
UNITED KINGDOM	1



20 Countries

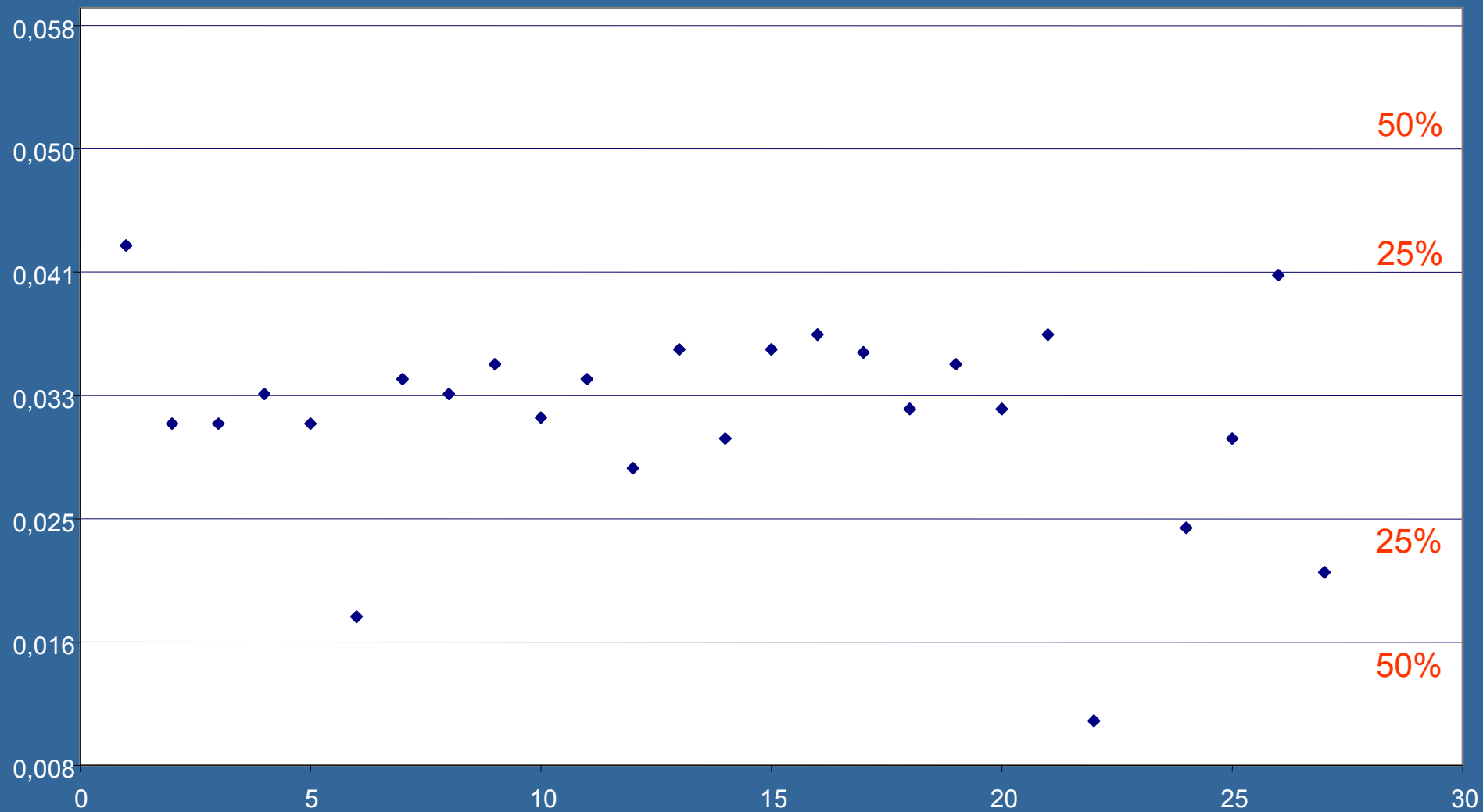
30 Laboratories



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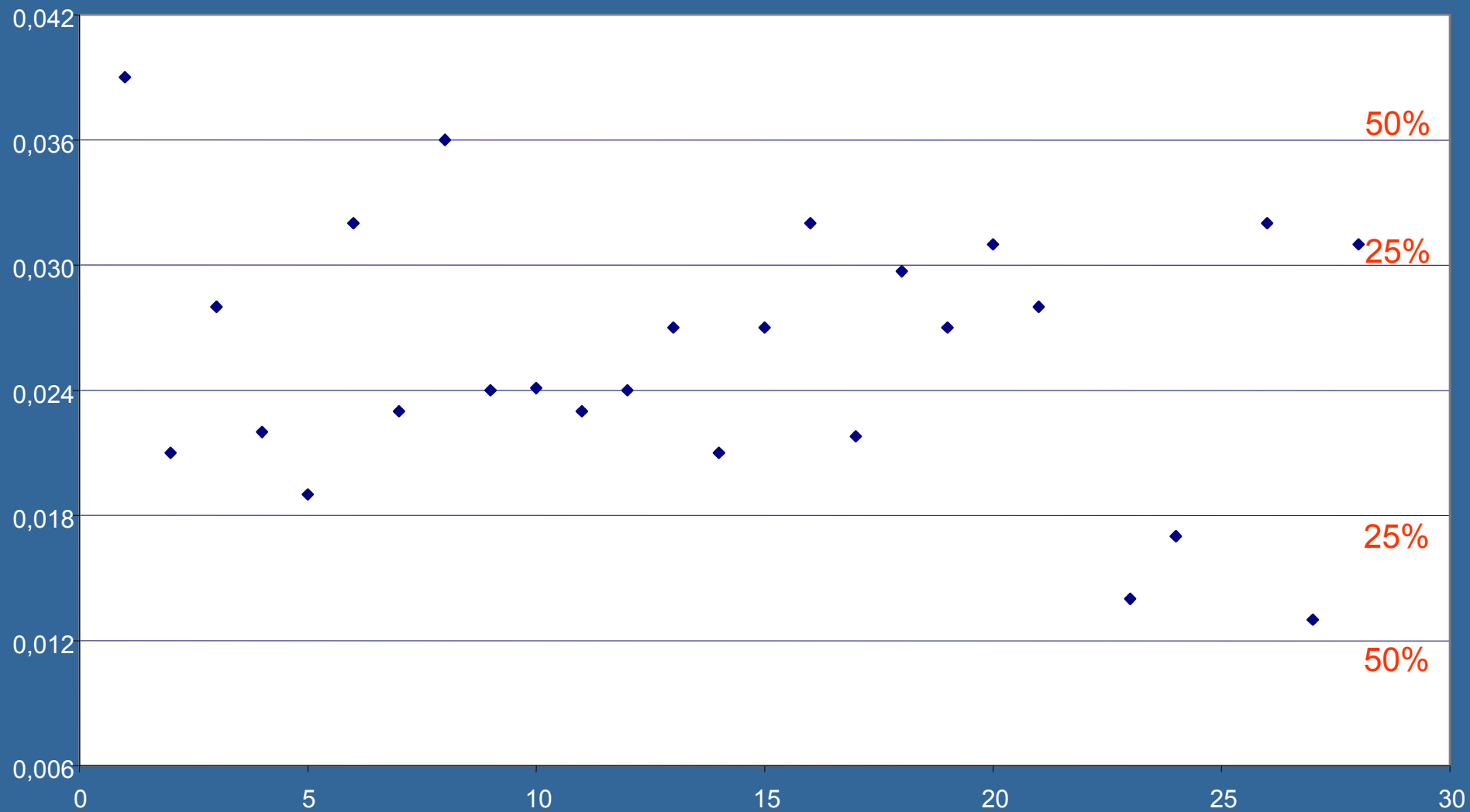
2007

Acetamidrid



2007

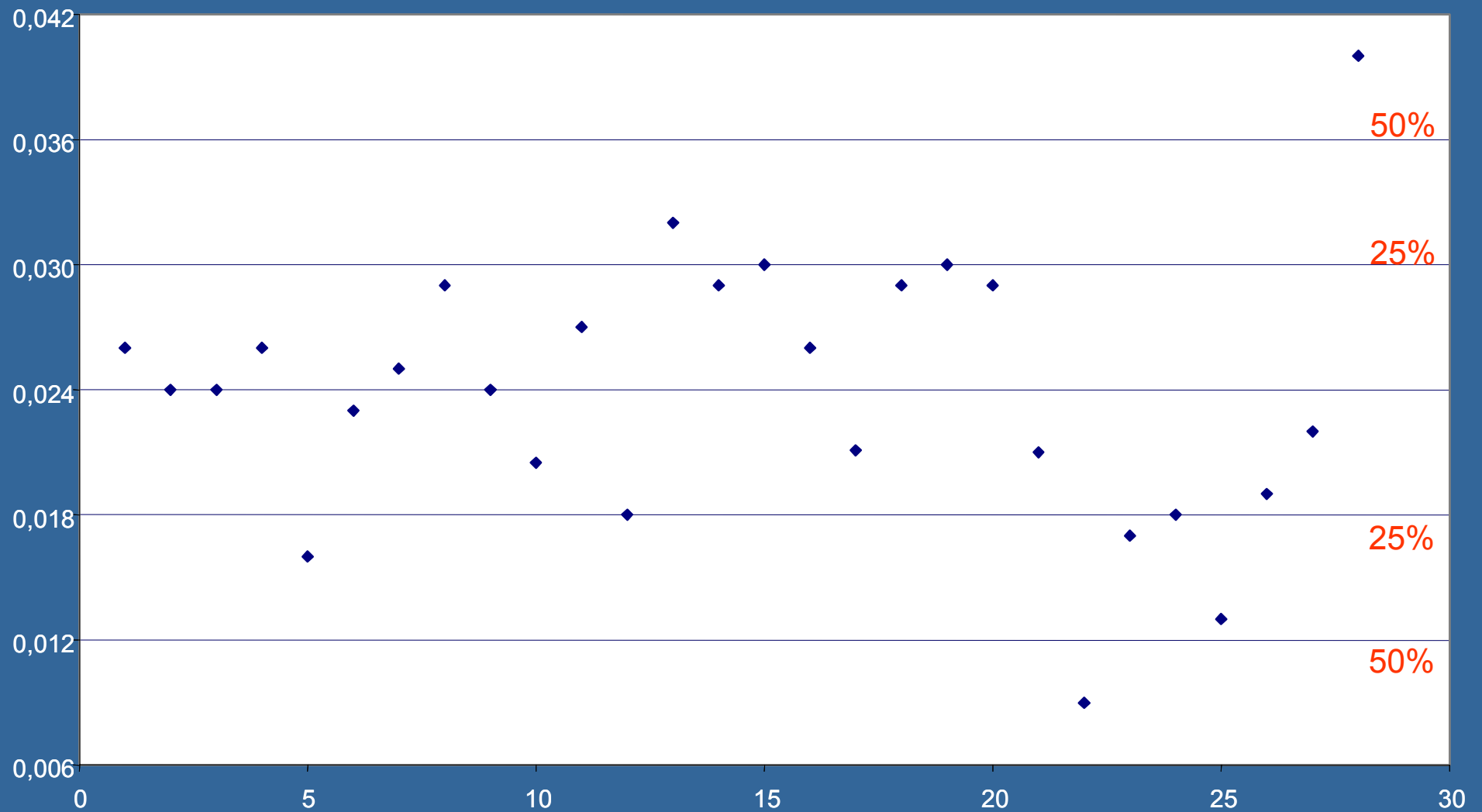
Carbaryl



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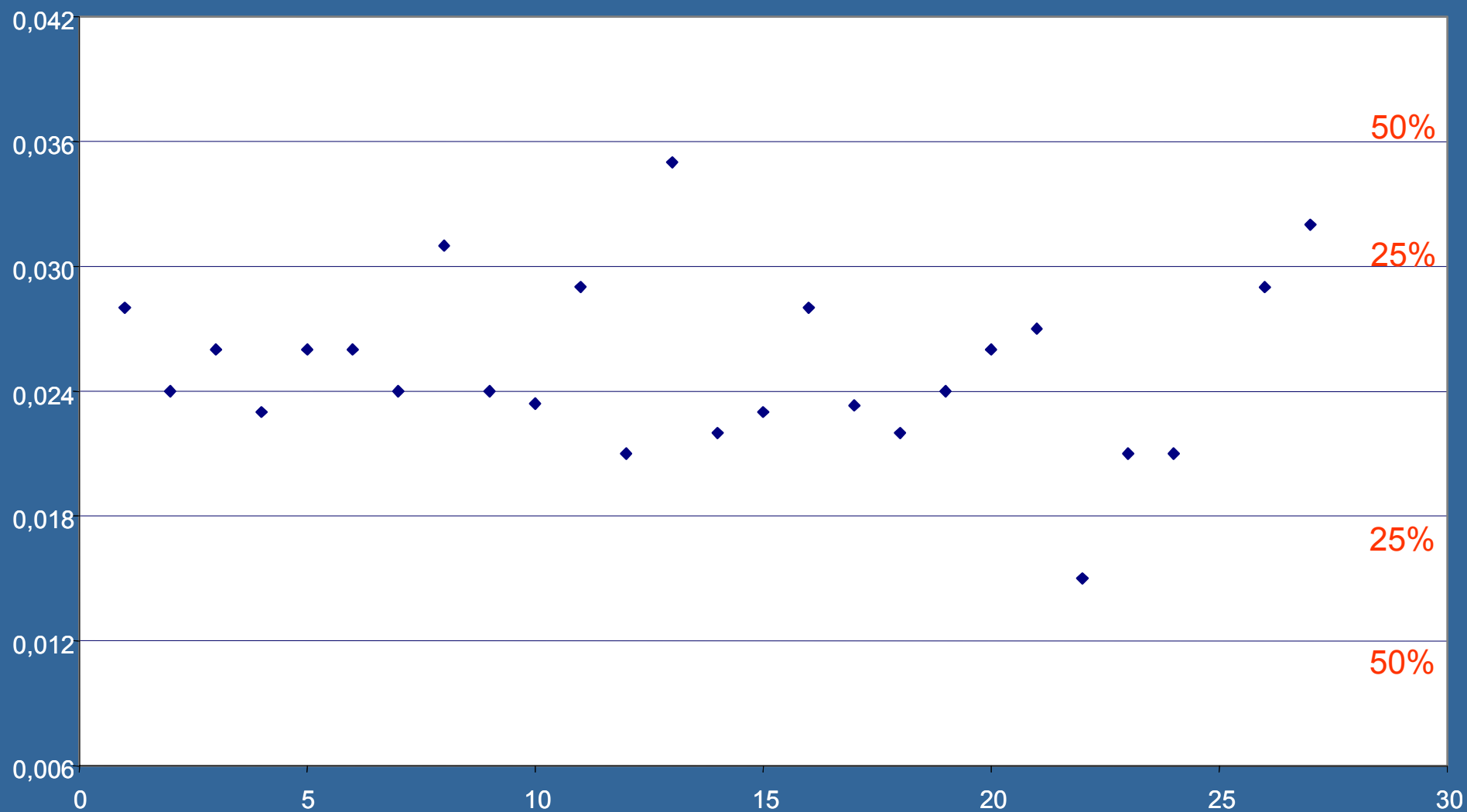
2007

Diazinon



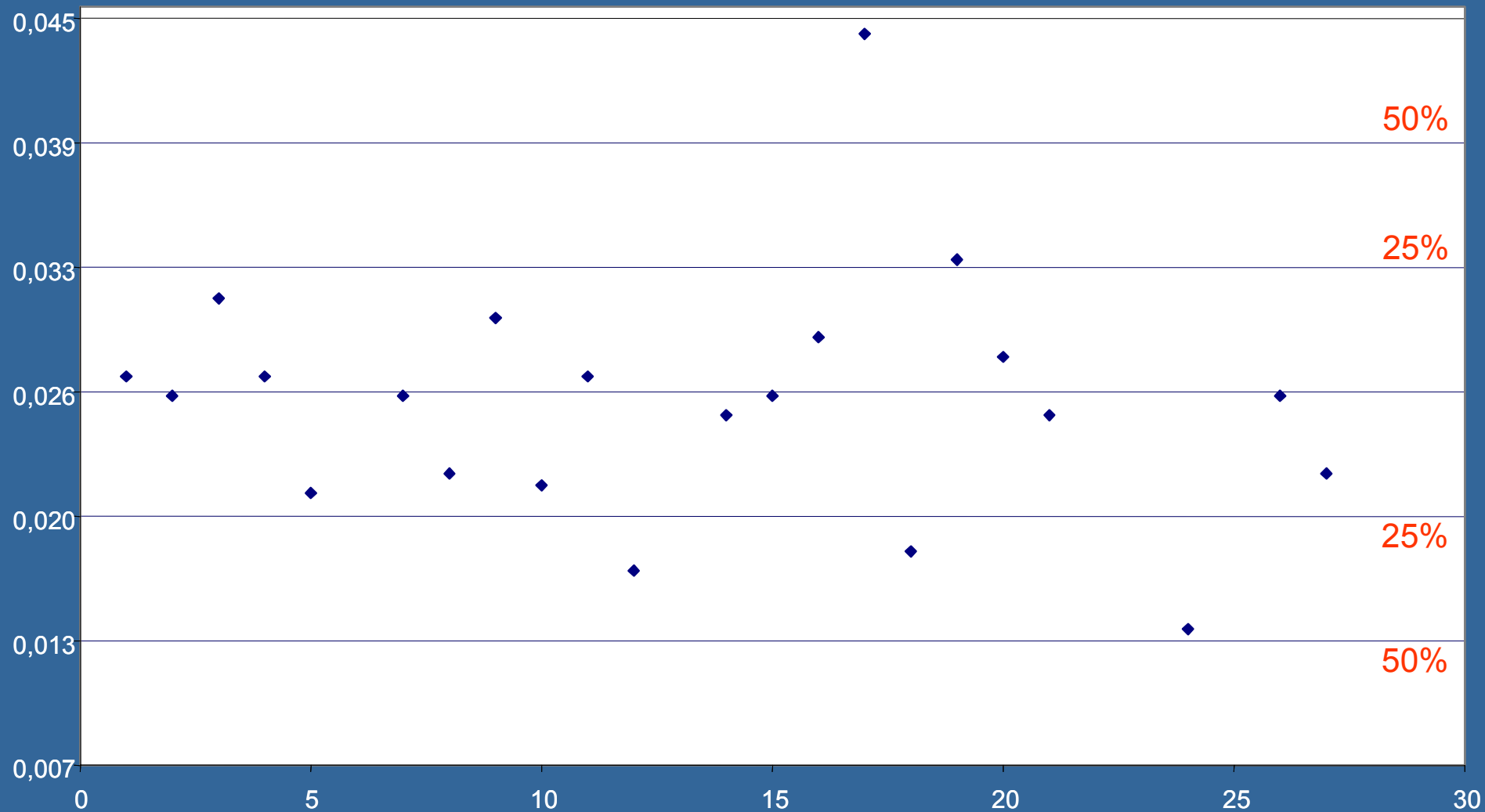
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Dimethoate



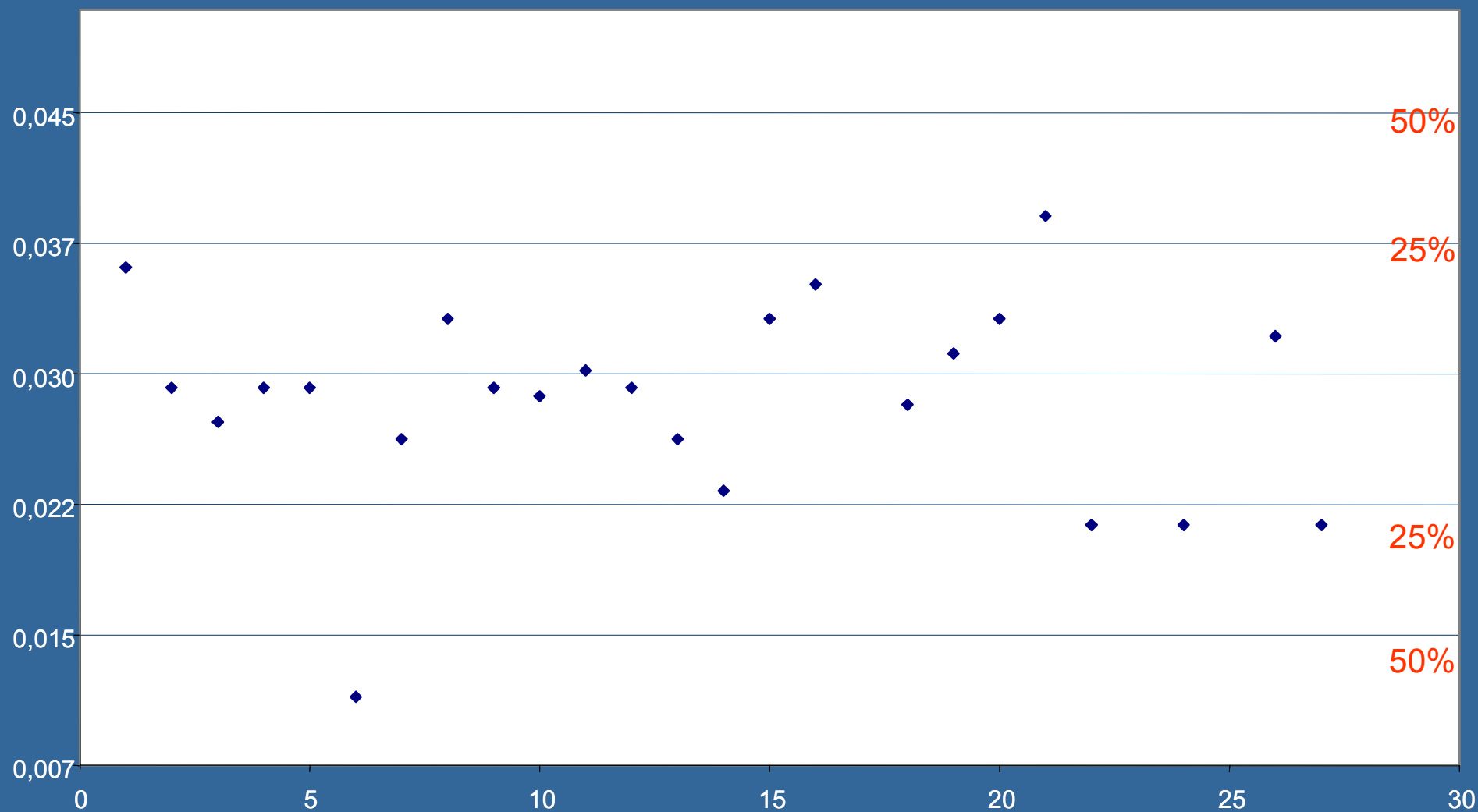
2007

Imazalil



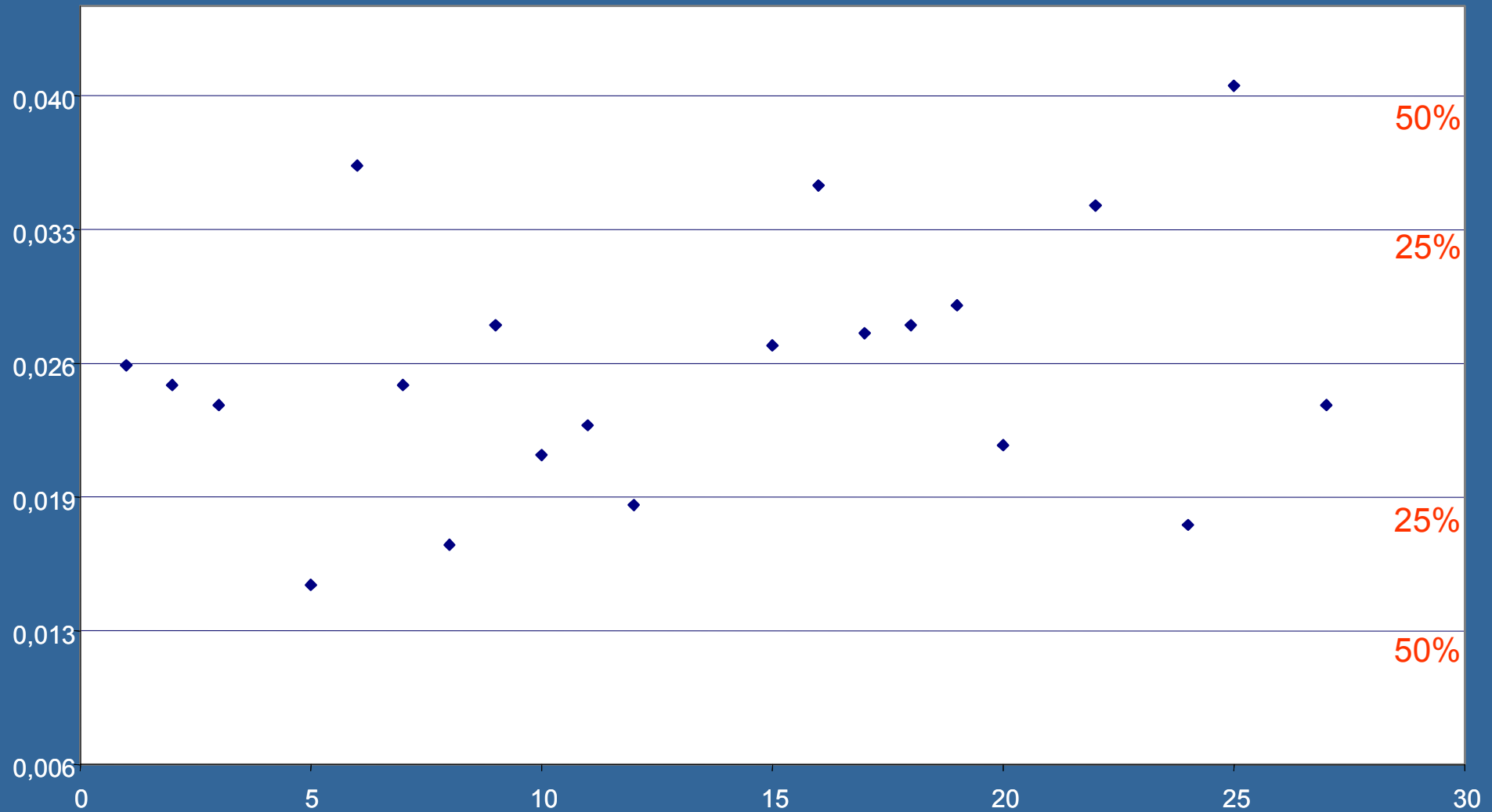
2007

Imidacloprid



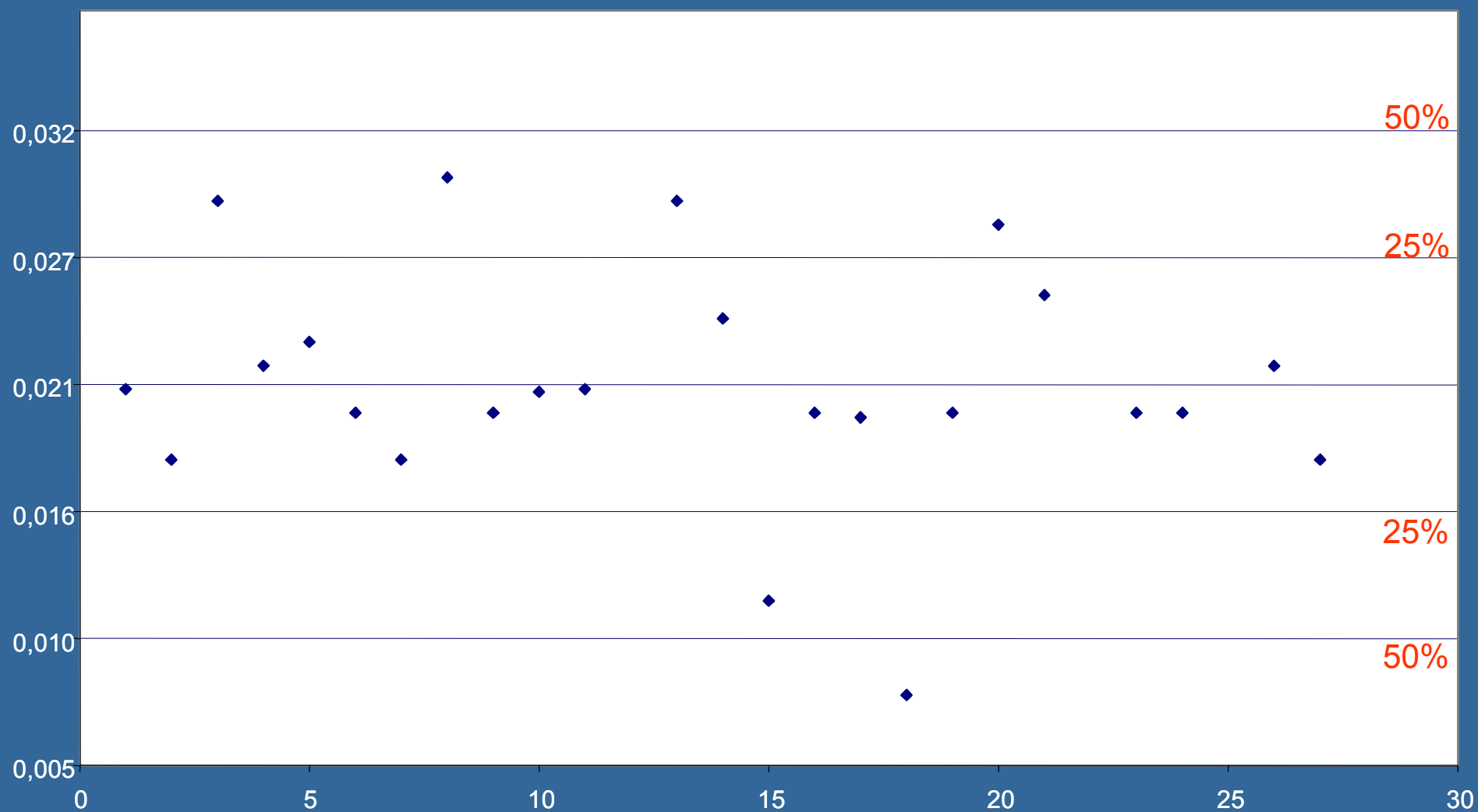
2007

Iprodione



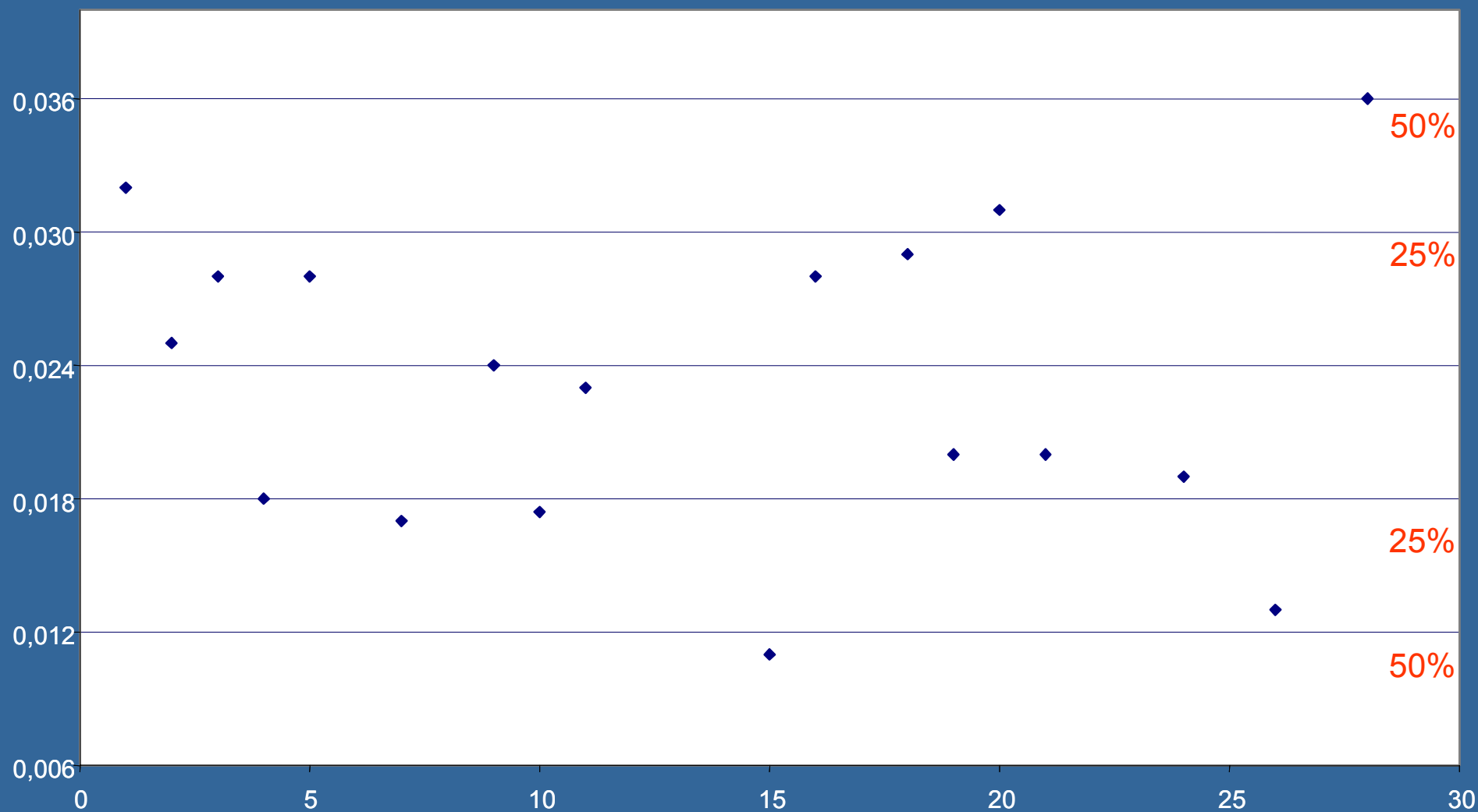
2007

Omethoate



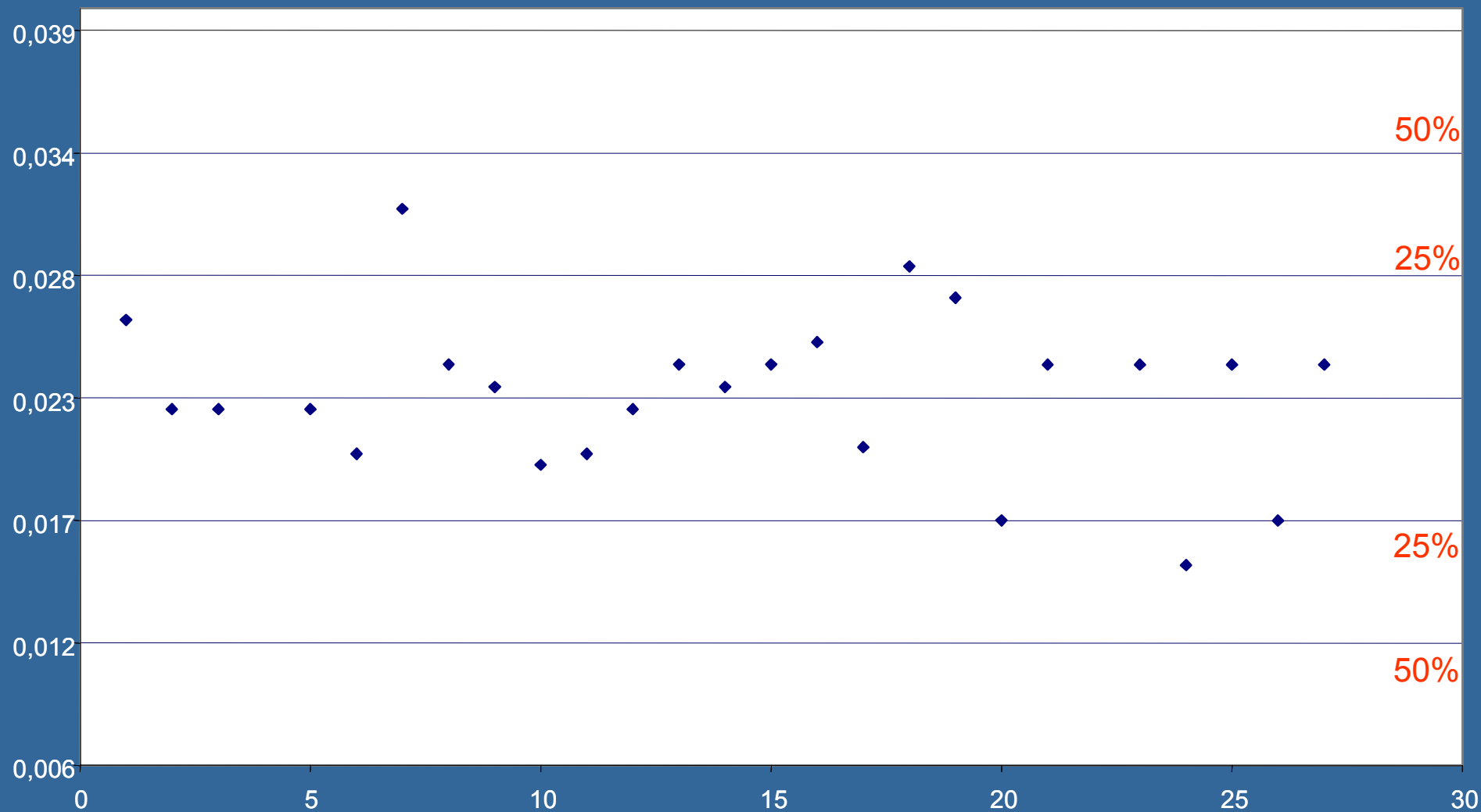
2007

Oxydemeton-methyl



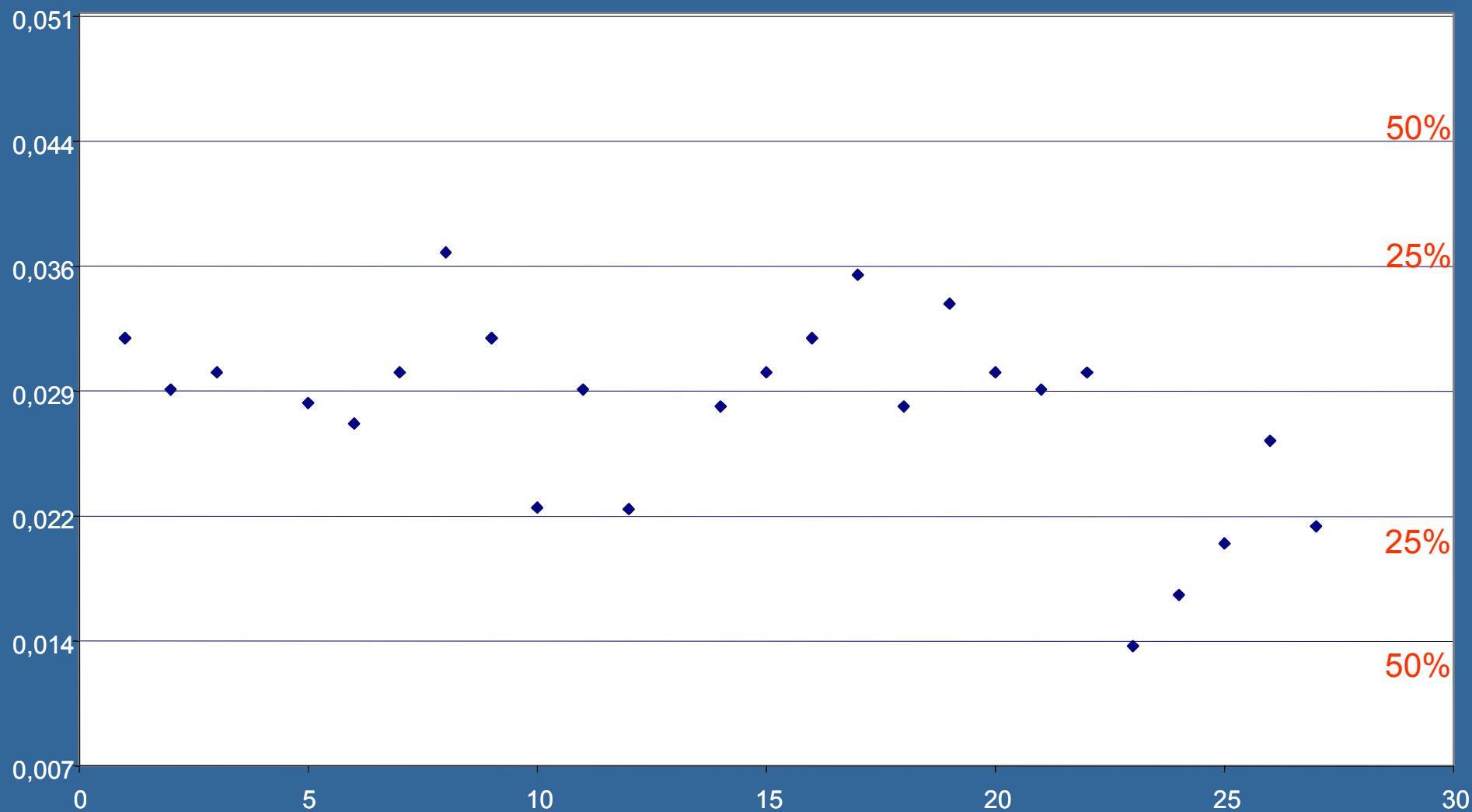
2007

Pyrimethanil



2007

Tetraconazole

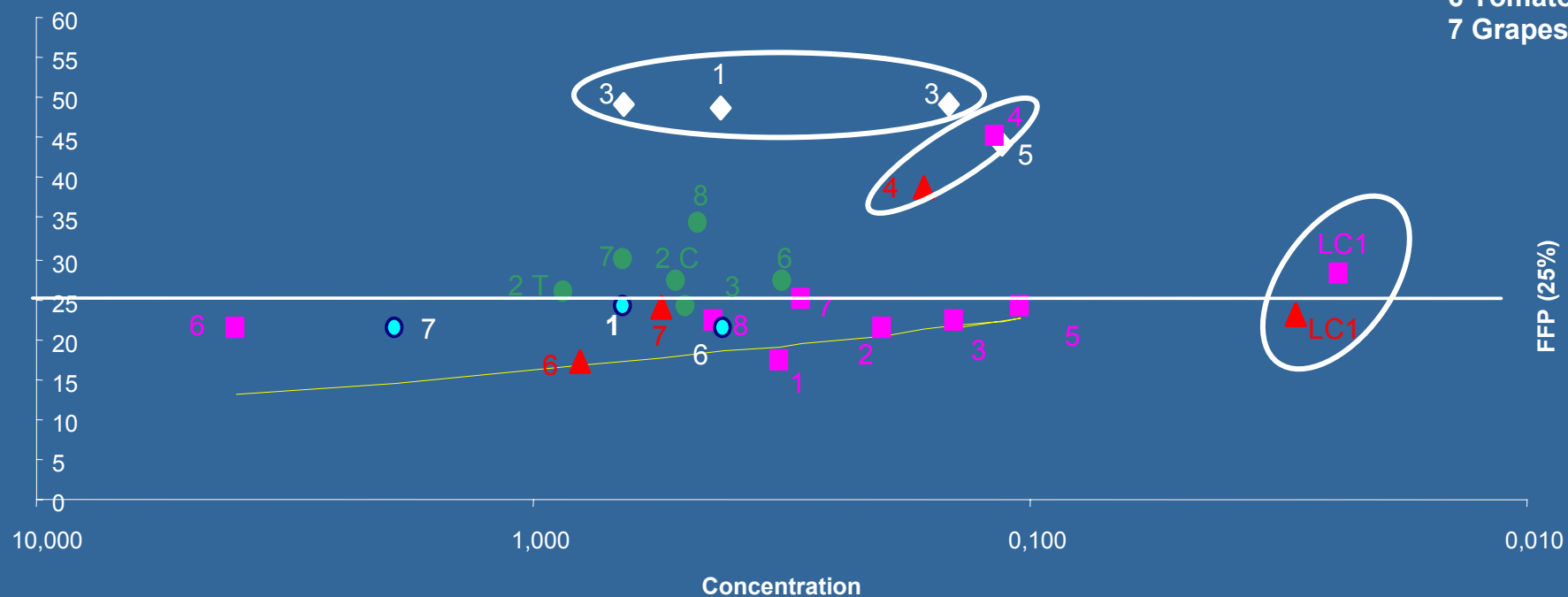


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Common Pesticides for all EUPT results

- 1 Peppers
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- 7 Grapes



◆ Acephate/Methamidophos ■ Diazinon ▲ Imidacloprid ● Procymidone ● Carbendazim/Thiabendazole — Horwitz

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